PB91-102053

# Technical Background Document and Response to Comments Resource Conservation and Recovery Act. Subtitle C-Hazardous Waste Management System. Section 3001: Identification and Listing of Hazardous Waste. Method 1311: Toxicity Characteristic Leaching Procedure (TCLP)

(U.S.) Environmental Protection Agency, Washington, DC

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The Agency received a total of 150 comments on the TCLP from 118 respondents. This document includes the response to commenters as well as serves as the background document.

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# TECHNICAL BACKGROUND DOCUMENT

# AND

# RESPONSE TO COMMENTS

Resource Conservation and Recovery Act
Subtitle C - Hazardous Waste Management System

Section 3001

Identification and Listing of Hazardous Waste

Method 1311
Toxicity Characteristic Leaching Procedure

(TCLP)

U.S. Environmental Protection Agency

Office of Solid Waste

April 1989

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# Introduction

On June 13, 1986, the USEPA proposed to amend its hazardous waste identification regulations under Subtitle C of the Resource Conservation and Recovery Act (RCRA) by expanding the Toxicity Characteristic (TC) to include additional contaminants (51 FR 21648). The agency proposed using a leaching test, the Toxicity Characteristic Leaching Procedure (TCLP), to determine if wastes should be classified as hazardous. Also, on July 9, 1986, (51 FR 24856) and September 19, 1986, (51 FR 33297) the Agency noted the availability of several other reports and studies containing additional data and information supporting use of the TCLP. The TCLP was promulgated on November 7, 1986, as part of the Land Disposal Restrictions Rule for Solvents and Dioxins (51 FR 40643-40652) and will be incorporated in the manual "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods" (SW-846) as Method 1311. In response to these Federal Register notices, the Agency has received a substantial number of comments from the American public. This document summarizes and responds to those comments which addressed the use of the TCLP in the Toxicity Characteristic Expansion rule.

The Agency received a total of 140 comments on the TCLP from 118 respondents. Twenty-two commenters addressed the TCLP as it was proposed in the January 14, 1986, Federal Register notice on the Land Disposal Restrictions rule, and eight of these later submitted comments on the June 13, 1986, Toxicity Characteristic proposal as well. Five organizations made presentations at the July 14, 1986, public hearing which addressed the proposed test, and three of these also submitted written comments. Seventy-five additional individuals or organizations submitted written comments on the TCLP as presented in the June 13, 1986, proposal. These commenters include 27 trade associations, 14 municipal sewer authorities, 44 other industries, 10 state or federal agencies (including Environment Canada), 9 hazardous waste management organizations, 4 consultants, 5 commercial laboratories, 3 individuals, and two public interest groups (the Environmental Defense Fund and the Natural Resources Defense Council). Twenty-seven additional comments were received after the August 12 deadline for comment submittal. These were reviewed and are included in this background document. The specific respondents are listed in Table 1.

On May 24, 1988, the EPA published proposed modifications to the TCLP (Method 1311) in the Federal Register (53 FR 18792-18798). The proposed changes included the use of a stainless steel cage in the bottle extractor, the addition of new equipment suppliers, and a revised method flowchart. The Agency received 22 comments on the proposed changes. These comments were reviewed and summaries and responses are included in Section VI of this Background Document. The specific respondents are listed in Table 2.

Each of the major issues regarding the TCLP is discussed in a separate section of this document. The issues are organized in a manner similar to that of the preamble to the June 13, 1986, Federal Register proposal with general issues addressed first, followed by comments on EPA's research relating to the development and evaluation of the leaching procedure, and then specific aspects of the extraction procedure and subsequent chemical analyses.

TABLE 1. COMMENTERS ON TCLP AS PROPOSED
JANUARY 14, 1986, AND JUNE 13, 1986

| Name of Commenter | LogNumber(s) of Comment(s) |
|---|---|
| CONSULTANTS: | |
| Battelle Pacific | TC0059 |
| Environmental Management and Engineering Inc. | TC0008 |
| Resource Consultants | TC0048 |
| University of Idaho | TC0011 |
| Total: 4 | |
| ELECTRIC UTILITIES: | |
| Detroit Edison | TC0111 |
| TVA | TCL017 |
| Total: 2 | |
| INDIVIDUALS: | |
| Brian B. Looney | TC0047 |
| Cate Jenkins | LDR024 |
| F. Winter and K. Farrell | TC0113 |
| Total: 3 | |
| INDUSTRIES: | |
| American Motors | TCL055 |
| American Water Works Service Co. | TC0087 |
| Atchison, Topeka and Sante Fe Railroad | TCL042 |
| Briggs and Stratton | TCL005 |
| Chrysler | TC0039 |
| Eastman Kodak Co. | TC0080 |
| Finnegan Mat | TC0102 |
| Ford Motor Co. | LDR006, TC0135 |
| General Battery Corp. | TC0037 |
| General Electric Co. | TCL026 |
| General Motors Corporation | TC0097 |
| HDR | TC0137, TCL007 |
| Heil Quaker Corp. | TC0010 |
| ITT Rayonier Inc. | TC0112 |
| Union Camp | TC0093 |
| Total: 15 | |

continued

TABLE 1. Continued

| Name of Commenter | LogNumber(s) of Comment(s) |
|---|---|
| LABORATORIES: | |
| Burmah Technical Services | PH0010 |
| Compuchem Laboratories | TC0045 |
| Enwright Laboratories | TC0063 |
| Lancy Environmental Services | TC0099 |
| Deuel and Zahray Laboratories | TCL113 |
| Total: 5 | |
| MINING/METALS PROCESSING: | |
| Alcoa | LDR016, PH0012 |
| Amax | LDR023, TC0140 |
| Asarco, Inc. | TC0098 |
| Auburn Foundry Inc. | TC0006 |
| Chemetco | TC0054 |
| J. R. Simplot Co. | LDR013 |
| Kaiser Aluminum | TC0119 |
| Reynolds Aluminum | TC0090 |
| Total: 8 | |
| MUNICIPAL SEWER AUTHORITIES: | |
| Cincinnati MSD | TC0096 |
| City of Colorado Springs | TC0003 |
| City of Indianapolis | TC0041, TC0051 |
| City of Memphis | TC0131 |
| City of New York DEP | TC0107 |
| East Bay Municipal Utility District | TCL016 |
| Hennepin Dept. of Environment and Energy | TC0070 |
| LA County Sanitation Dist. | TC0128 |
| Metro Denver Sewage Disp. Dist. | TC0139 |
| Metro Seattle | TC0130 |
| Metropolitan Waste Control Commission of St. Paul | TC0120 |
| MSD of Greater Chicago | LDR003, TCL008 |
| Narragansett Bay Commission | TC0094 |
| Northeast Ohio Regional Sewer District | TC0089 |
| Total: 14 | |

continued

TABLE 1. Continued

| Name of Commenter | LogNumber(s) of Comment(s) |
|---|---|
| PETROLEUM/CHEMICAL COMPANIES: | |
| Air Products and Chemicals Inc. | TC0117, TCL092 |
| American Cyanamid Company | TC0035 |
| Amoco Corporation | TC0058 |
| Chevron | TC0060, TCL077 |
| Colorado Interstate Gas Co. | TC0091 |
| Conoco | TC0116, TCL066 |
| Diamond Shamrock | LDR010, TCL013 |
| DOW Chemical | LDR021, TC0057, TCL088 |
| El Paso Natural Gas | TC0052 |
| Kaiser Chemicals | TC0108 |
| Lubrizol Corporation | TC0088 |
| Mobil Oil Corp. | LDR027 |
| Questar Corp. | TC0101 |
| Shell Oil Company | TC0073 |
| Standard Oil Company | TC0056 |
| Syntex Agribusiness Inc. | LDR017 |
| Texaco | LDR014 |
| Unocal Corp. | TCL082 |
| Vulcan Chemicals | TC0138 |
| Total: 19 | |
| PUBLIC INTEREST GROUPS: | |
| Environmental Defense Fund | TC0076 |
| Natural Resources Defense Council | TCL112 |
| Total: 2 | |
| GOVERNMENT AGENCIES: | |
| Commonwealth of Virginia | TC0067 |
| Department of Energy | TCL019 |
| Environment Canada | TC0005 |
| Maryland Waste Mgt. Admin. | TC0127 |
| Minnesota Pollution Control Agency | TC0126 |
| NEIC | TC0071 |
| New Hampshire DPHS | TC0103 |
| New Jersey Dept. of Environmental Protection | TC0007 |
| NY State Dept. of Environmental Conservation | TC0066 |
| Texas Water Commission | TC0053 |
| Total: 10 | |

continued

TABLE 1. Continued

| Name of Commenter | LogNumber(s) of Comment(s) |
|---|---|
| TRADE ASSOCIATIONS: | |
| American Mining Congress | LDR005 |
| American Petroleum Institute | LDR012, TC0118 |
| Association of Metropolitan Sewerage Agencies | TC0043, PH0003 |
| Assoc. of State and Territorial Solid Waste Mgt. Officials | LDR020 |
| Association of American Railroads | TC0074 |
| Can Manufacturers Institute | LDR009 |
| Chemical Manufacturers Association | LDR031, TC0002, TC0081 |
| Government Refuse Collection and Disposal Assoc. | TC0030 |
| Halogenated Solvents Industry Alliance | TC0083 |
| Hazardous Waste Treatment Council | LDR030, PH0001 |
| Institute of Scrap Iron and Steel, Inc. | TC0105 |
| International Fabricare Institute | TC0106 |
| Interstate Natural Gas Association of America | TC0124 |
| Leather Industries Research Lab | LDR001, TC0024 |
| Michigan Chamber of Commerce | TC0123 |
| Michigan Manufacturers Association | TC0064 |
| National Council of the Paper Industry for Air & Stream Improvements | LDR025, TC0077, TC0123 |
| National Electrical Manufacturers Assoc. | TCL060 |
| National Lime Association | LDR011 |
| National Paint and Coatings Assoc. | TCL025 |
| Pharmaceutical Manufacturers Assoc. | TCL084 |
| Porcelain Enamel Institute, Inc. | TC0044 |
| Rocky Mountain Oil and Gas Association, Inc. | TC0129 |
| Society of the Plastics Industry | TCL095 |
| Specialty Steel Industry of the US | TC0078 |
| Synthetic Organic Chemical Manufacturers Assoc. | TCL049 |
| Utility Solid Waste Activities Group | LDR004, TC0072 |
| Total: 27 | |
| WASTE MANAGEMENT COMPANIES: | |
| Browning-Ferris Industries | TC0065 |
| Cecos International | TC0079 |
| Chem Met Services | TCL023 |
| Chemical Waste Management, Inc. | TC0062, TCL036 |
| Envirite Corporation | TC0125 |
| Envirosafe Services, Inc. | TC0095 |
| Penberthy Electromat Company | PH0008 |
| Waste Management Inc. | LDR028 |
| Wayne Disposal, Inc. | TC0022 |
| Total: 9 | |
| Grand Total: 118 commenters | |

TABLE 2. COMMENTERS ON MAY 24, 1988 PROPOSED MODIFICATIONS

| Name of Commenter | LogNumber(s) of Comment(s) |
|---|---|
| PETROLEUM/CHEMICAL COMPANIES: | |
| Conoco, Inc. | TCA00010 |
| Eastman Kodak | TCAL0002 |
| Lubrizol Corporation | TCA00011 |
| Monsanto Company | TCA00009 |
| Vulcan Material Company | TCA00018 |
| LABORATORY: | |
| Analytical Testing and Consulting Services | TCA00006 |
| MINING/METALS PROCESSING: | |
| Aluminum Company of America | TCA00001 |
| ASARCO, Inc. | TCA00005 |
| Chemetco | TCA00021 |
| TRADE ASSOCIATIONS: | |
| Association of American Railroads | TCA00007 |
| Chemical Manufacturers Association | TCA00016 |
| Institute of Chemical Waste Management | TCA00017 |
| Institute of Scrap Recycling Industries, Inc. | TCA00019 |
| Lead Industries Association, Inc. | TCAL0001 |
| National Council of the Paper Industry for Air and Stream Improvements, Inc. | TCA00003 |
| Secondary Lead Smelters Association | TCA00015 |
| Steel Structures Painting Council | TCA00004 |
| Society of the Plastics Industry | TCA00014 |
| Steel Bar Mills Association | TCA00008 |
| Utility Solid Waste Activities Group | TCA00013 |
| ELECTRIC UTILITIES: | |
| Pacific Gas and Electric Company | TCA00020 |
| WASTE MANAGEMENT COMPANIES: | |
| Chemfix Technologies, Inc. | TCA00002 |
| TOTAL: 22 Commenters | |

## I. General Comments

### A. Relationship to EP

Thirty-three respondents expressed opinions on the relative merits of the EP and TCLP tests and were almost evenly divided on the subject. Eighteen comments asserted that the TCLP is an improvement over the EP, while fifteen stated that the new test provides no advantage or that the EP should be retained.

One comment favoring the TCLP asserted that the proposed test represents a substantial improvement over the EP toxicity test, while another stated that any improvement over the current EP test is welcome. Most comments were more qualified in their support. One claimed that the current extraction procedure (EP) is inadequate, and that while the proposed TCLP may represent some degree of improvement over the EP, there are still serious doubts about its validity and replicability. Other comments expressed similar opinions.

Nine comments asserted that the TCLP provides no advantage over the current EP and that, even though the current EP test has some serious weaknesses, there is little evidence that the TCLP is an adequate substitute. Six comments stated that the EP should be retained, at least for metals and non-volatile organics.

As discussed in the preamble, the Agency believes that the TCLP is needed in order to permit the expansion of the Toxicity Characteristic to include volatile organics, and also represents an improvement over the EP. Previous experience showed that the recovery of volatiles with EP equipment was poor (S-Cubed, 1986c) and thus the EP is unsuitable for estimating the leaching potential of volatile chemicals. The TCLP procedure is also simpler to perform than the EP for metals and semi-volatile organics. Operational simplifications include eliminating the requirement for continuous pH adjustment and replacing the 0.45 micron membrane filter with the 0.6 - 0.8 micron glass fiber filter to aid liquid/solid separation. Specifying only one acceptable means of agitating the samples also removes a source of variability in the test. (USEPA, 1986a)

## II. Research Supporting Leaching Procedure Development

### Background

The Agency's research which led to the development of the proposed TCLP consisted of three phases. The first phase was designed to narrow the range of possible leaching procedures, and the second phase consisted of more extensive evaluation of a smaller selection of tests. This resulted in choosing one procedure as the draft TCLP. The third phase involved precision and ruggedness evaluation of the selected method. Phases I and II are discussed in this section of the background document while Phase III is addressed in the following section.

In designing the research program, the Agency used the same model and assumptions used in developing the EP test; namely, co-disposal of industrial waste with municipal waste in a sanitary landfill. In conducting the tests for the Agency, the U.S. Department of Energy's Oak Ridge National Laboratory (ORNL) began Phase I with field lysimeters which were filled with domestic and commercial refuse. This was done in order to generate a municipal waste leachate which would be representative of the mismanagement scenario. The generated leachate was used to extract four industrial solid wastes in pilot scale leaching columns, and the concentrations of various organic and inorganic parameters in the leachate were measured over time. (Francis et al., 1984)

Next, target concentrations were developed for each constituent of interest. These concentrations were based on data from the pilot columns and were compared with the results of a variety of laboratory leaching procedures. The lab tests which were evaluated included both batch and column procedures using four leaching media and four media-to-waste ratios, in addition to the existing EP test and a sequential batch leaching procedure. The two laboratory procedures which most closely represented the results of the pilot scale columns (according to statistical analysis and other factors) were further evaluated in Phase II.

Both procedures selected for Phase II were batch tests which utilize a 20:1 liquid-solid ratio. One used a 0.5 N pH 5 sodium acetate buffer solution as the extraction fluid while the other used distilled, deionized water. Seven additional industrial solid wastes were exposed to municipal waste leachate in the pilot scale columns (as in Phase I). They were also subjected to the EP test and the two selected laboratory procedures. The results were compared, analyzed statistically, and evaluated according to EPA's stated objectives for the test. Then a draft TCLP was developed. (Francis and Maskarinec, 1986) The Agency circulated the draft to interested groups outside the Agency for comment and evaluated it for precision and ruggedness during Phase III.

## A. General Comments

The Agency received forty general comments from 17 different commenters on the research which led to the development of the leaching procedure. These comments were submitted by public interest groups, government agencies, an individual, a utility company, a manufacturer, various trade associations, municipalities, and oil, gas, and chemical companies. All comments were generally critical. Several comments asserted that the test method should be based on both intrinsic properties of the waste and a fundamental understanding of the mechanisms of leaching instead of empirical observations made by ORNL. A number of comments stated that the TCLP does not take into account the time-dependent nature of the leaching process. Other comments, including those from oil companies and municipalities, voiced the concern that some specific types of wastes were not included in the method development studies, particularly multi-phase or oily wastes and municipal sewage sludge. For this reason, the comments asserted that evaluation of eleven samples was insufficient to support EPA's conclusion that further testing will not result in significant changes in the composition of extracted fluids. Several comments questioned whether the leachate generated in the field lysimeters was actually representative of leachate from municipal sanitary landfills. Finally, several comments made general remarks about the inadequacy of the research supporting the method development.

The Agency supports the use of the TCLP for identification of hazardous wastes and believes that the Oak Ridge research demonstrates the applicability of the test for this purpose. Insufficient information is available at this time to develop identification criteria based on intrinsic waste properties and fundamental mechanisms of leaching. The Agency has therefore elected to devel an empirical test to evaluate potential hazard. The Agency believes that the eleven wastes used in the Oak Ridge research are representative of a wide variety of waste types, with the exception of highly alkaline wastes as discussed elsewhere (USEPA, 1986a). These and other issues related to the Oak Ridge research are discussed in greater detail in the following sections.

## B. Oak Ridge National Laboratory Testing: Phase I

Thirteen comments addressed Phase I of the ORNL research, and all were critical of the initial development work for the TCLP. Five of these comments criticized the generation of municipal waste leachate for the study. In particular they noted that, since the lysimeters were operated for only two months, the generated leachate cannot be considered representative of leachate from actual landfills, which develops over the course of several years. One comment asserted that the conditions represented in Phase I cannot be considered as the worst possible case for three reasons: industrial wastes and small quantities of hazardous wastes were not included in the lysimeters for the generation of leachate; the time of leaching was too short to generate leachate of maximum aggressiveness; and the potential interactions of the industrial wastes were ignored.

The Agency notes that the waste in the Oak Ridge lysimeters was allowed to develop for three months before leaching with distilled water began. This development period allowed biological degradation to become established in the waste. Distilled water was then pumped through the lysimeters for two months to allow the composition of the leachate to reach a steady-state before leaching of the industrial wastes was initiated (Francis et al. 1984). The EPA's intent was to model the early stages of landfill decomposition. Leachate is believed to be most aggressive in its mobilizing ability (acidity and complexation ability) during the first few years of landfill life (Kimmell and Friedman, 1984). Construction of the lysimeters with the three month development time prior to the start of leaching was used as a reasonable approximation of conditions during what was believed to be the most aggressive stage of landfill life.

The Agency considered several mismanagement scenarios and decided on co-disposal of hazardous waste with municipal refuse in a sanitary landfill as the basis for the TCLP (as for the EP (USEPA, 1986a)). This scenario represents a reasonable form of mismanagement with respect to potential for ground water contamination. Available data indicate that few organic solvents or other industrial liquid wastes are disposed of in municipal sanitary landfills (USEPA, 1986a), and as a result of stricter regulatory controls even fewer of these types of wastes will be so disposed in the future. The test is therefore intended to approximate the leaching action of low molecular weight carboxylic acids generated by the decomposition of household and commercial municipal wastes in a sanitary landfill (USEPA, 1986a). Such landfills are usually constructed in cells or lifts separated by layers of soil. Interactions of industrial wastes and mixing of industrial and municipal wastes are assumed to be minimal.

One comment questioned the relationship between the liquid-to-solid ratio in a laboratory procedure and the actual time of leaching in the field. The comment pointed out that the effect of the liquid to solid ratio on the concentrations obtained in a batch leach test depends on the relative aggressiveness of the extraction fluid and the total quantity of contaminant available in the waste.

The Agency agrees that the effect of liquid-to-solid ratio does in fact depend on many factors, including waste composition, alkalinity, and extractant. The relationship between liquid-to-solid ratio in the laboratory and the actual time of leaching in the field also depends on site-specific factors such as the depth of the landfill and the local precipitation patterns. Therefore, the relationship should be interpreted broadly in terms of relative differences and not absolute values (Kimmell and Friedman, 1984).

One comment stated that the constituent concentrations in the leachate from the Oak Ridge industrial waste columns are higher than what would be found in the field since the municipal waste leachate was used to leach columns containing 100% industrial waste, instead of a mixture of industrial waste with municipal waste.

The Agency again points out that the scenario modeled by the TCLP is the same scenario modeled by the EP. The specific assumptions are that industrial waste is disposed of in a sanitary landfill along with municipal waste in a ratio of 5:95 and that the leachate produced is primarily a function of the biological decomposition of the municipal refuse (USEPA, 1986a). Municipal and industrial wastes are not typically mixed in landfills but disposed of in separate cells or lifts.

The development of target concentrations for evaluating the candidate leaching procedures was also criticized. The comment asserted that the maximum leachate concentration would be more conservative than the average concentration used in the study and that the conservative approach to this situation would be to assume a continuous release at maximum leachate concentration from an infinite source.

The Agency disagrees with this comment. The TCLP is intended to model chronic (long-term) exposure to contaminants in drinking water. EPA has no data to demonstrate that a continuous release of contaminants at the maximum leachate concentration occurs in the real world. Such an assumption would not constitute a reasonable worst case, but an absolute worst case.

Two comments criticized the design and evaluation of the laboratory leaching tests, stating that batch and column tests should not be compared, since batch tests can reach a steady state condition while column leach tests always operate under dynamic conditions. Also, no information on how well the field lysimeters actually simulate landfill conditions was presented in the proposed rule.

The Agency points out that the same liquid-to-solid ratios were applied in both the column tests and the batch tests, thereby providing a basis for comparison. Regarding data on actual landfill leachate composition, few sanitary landfills had (or have) leachate collection systems and so at the time of method development little information was available from tests of actual leachate composition. Available information from laboratory and pilot-scale investigations are discussed in the reports on the Oak [illegible]ge research (Francis et al, 1984; Francis and Maskarinec, 1986).

Other comments criticized the method development work for not including specific wastes, such as oily and multiphasic wastes and municipal sewage sludge.

The Agency stated previously that it is not necessary to test every possible waste during method development provided that the wastes tested represent the universe of waste types. One of the four wastes used in the Phase I tests was in fact an API separator sludge from a petroleum refinery mixed with refinery incinerator ash (Francis et al, 1984). Additional studies are underway on the use of the TCLP on oily wastes (see Sections IV.A and V.J of this document).

## C. Oak Ridge National Laboratory Testing: Phase II

The Agency received nine critical comments in this area. One comment criticized the selection of leaching procedures for development. It asserted that a narrow range of extraction media was studied, none of which was significantly different from the current EP test. It also asserted that the Agency's rationale for the media selection was incomplete and uninformative. According to this comment, the Agency also failed to consider complexation or the effects of co-solvents on mobility, and the solubility of non-polar organic compounds when selecting the extraction fluids to be evaluated. Two comments also made the point that two of the fluids tested ("$CO_2$ saturated, deionized, distilled water" and "deionized, distilled water") are not different since water normally reaches equilibrium with $CO_2$ in the atmosphere. Also, the acetate buffer solution is only a minor modification of the existing EP.

The Agency responds that in the disposal scenario modeled by both the EP and the TCLP, the leaching medium is composed of precipitation onto the landfill combined with decomposition products from municipal wastes. These decomposition products consist primarily of carboxylic acids, one of the most common of which is acetic acid. There is no reason for the extraction fluid for the TCLP to be radically different from that used in the EP, since the tests are both based on the same assumptions. The acetate buffer was selected over the acetic acid used in the EP to make the test easier to perform. This rationale is discussed in several reports on the test method development (Francis et al, 1984; Kimmell and Friedman, 1984; Francis and Maskarinec, 1986; USEPA, 1986a).

The assumptions of the disposal scenario preclude the presence of significant concentrations of solvents in the extraction medium. Carboxylic acids, which are complexing agents, made up 60% to 80% of the TOC of the lysimeter leachate (Francis et al, 1984) so the complexing effect of these compounds is accounted for. Comparison of the candidate extraction fluids with the lysimeter leachate indicates that the effects of complexation and solubility are adequately represented by the laboratory procedure.

Another comment criticized the limited number of wastes used in Phase II. Only two of the original eleven wastes were used in the second phase of the study to validate the TCLP. According to this comment, the limited number of wastes in the study does not validate or even recommend the test for characterizing all solid wastes.

The Agency points out that four industrial solid wastes were used in Phase I and seven additional wastes were tested in Phase II. These eleven wastes represented a wide range of waste composition and characteristics. Phase I wastes consisted of heavy ends and column bottoms from the production of tri- and perchloroethylene, a paint production sludge, a mixture of refinery incinerator ash and API separator sludge, and an electroplating wastewater treatment sludge (Francis et al., 1984). The wastes used in Phase II were an industrial wastewater treatment sludge, ammonia lime still bottoms, a mixture of organic solvent and sludge, a mixture of organic solvent and vermiculite, a mixture of still bottoms and wastewater treatment sludge, a mixture of still bottoms and vermiculite, and a utility fly ash (Francis and Maskarinec, 1986). It is not necessary to test every waste if the wastes which are tested adequately represent the universe of waste types. Phase III of the TCLP development consisted of tests to evaluate intra- and inter-laboratory ruggedness and precision. Two wastes were used in the Phase III evaluation (USEPA, 1986a).

Another comment expressed some confusion about references to the "lysimeter field model" and the ability of different leachate media to duplicate "field results" since there were no leaching tests of industrial wastes in field lysimeters.

The Agency points out that the industrial wastes were exposed to lysimeter leachate in pilot scale columns, 38.7 cm in diameter by 30.5 cm high, which were located adjacent to the lysimeters (Francis et al, 1984). These tests are referred to as field tests to distinguish them from the laboratory tests.

## D. Statistical Treatment of Data

One comment addressed in detail both the multivariate analysis and other aspects of the statistical treatment of data from the ORNL studies. It asserted that the Mahalanobis distance statistic (used to evaluate the various extraction fluids) has a highly non-normal distribution and is thus unsuitable for a multivariate type of analysis. Specifically, using such skewed data violates the assumptions behind the multivariate analysis procedure and may result in false conclusions drawn from the analysis. Also, significant waste/media interactions confound the multivariate analysis and preclude the ability to make comparisons across all wastes. When comparing test results on a waste-by-waste basis, there is no evidence for a single, clearly superior extractant. Another comment asserted that a review of the ORNL data shows that the best long-term average predictor is a test which uses distilled water as the leaching medium.

The Agency anticipated criticism of the statistical analysis of the Oak Ridge data, and attempted to address these critics in advance by submitting the data to several different analyses. The results of these analyses were discussed extensively in the TCLP background document prepared for the June 13, 1986, proposal (USEPA, 1986a). The Agency recognizes that no single leaching procedure will be able to accurately predict concentrations of all compounds in all waste matrices. The intent of the investigation was to determine what procedure provides the best representation over all conditions. In order to evaluate this, the results must be compared across all waste types. The Agency points out that other factors were considered in development of the TCLP besides the results of the statistical analysis. These factors include practicality, ease of operation, cost, reproducibility, and applicability to both organic and inorganic contaminants.

The Agency is continuing to investigate the use of distilled water as the extractant for testing of volatile organics. Previous research has shown that the leaching of organics is not affected by changes in the leaching fluid, whereas the composition of the leaching fluid is important for the leaching of inorganics (Warner et al., 1981). It is also desirable to use the same leaching fluid for all segments of the test. The TCLP as finalized for use in the Toxicity Characteristic requires an acetate buffer leaching fluid for most wastes and an acetic acid leaching fluid for highly alkaline wastes.

Another comment stated that evaluating the batch tests on the basis of only one organic component, naphthalene, does not validate the procedure.

Naphthalene was not the only organic compound evaluated in development of the TCLP. Other compounds included in the Phase I evaluation were di- and trichloroethylene, di- and trichloroethane, toluene, xylenes, and hexachlorobutadiene (Francis et al, 1984). Phase II evaluation included 10 inorganic and 12 organic compounds (Francis and Maskarinec, 1986).

One comment pointed out that the accuracy of the proposed acetate buffer is extremely poor for a very important class of chemicals (i.e. chlorinated compounds). On an average, the acetate buffer extracted only 22% of the target concentrations for this class of compounds in the Phase II tests. The comment raised the concern that chlorinated organics will be under-represented by the TCLP.

The Agency replies that the poor extraction of chlorinated organics (generally classified as volatiles) in Phase II of the research was noted. These results led the Agency to conclude that volatiles were being lost as a result of the air pressure filtration and the headspace within the bottle extractor. Consequently, the Agency determined that a different device was needed to improve the recovery of volatile compounds. Thus, the Zero Headspace Extractor (ZHE) was developed to minimize the loss of volatiles.

## III. Research Supporting Leaching Procedure Development: Ruggedness and Precision Evaluation

Phase III of the EPA's research program consisted of evaluating the proposed procedure for precision and ruggedness on an intra-laboratory basis and conducting an inter-laboratory collaborative study. The precision evaluation was performed by two laboratories which used two industrial wastes. Precision of the test for non-volatile components was judged acceptable, while the evaluation of precision for volatile components was hampered by problems with equipment and laboratories and changes in the test protocol. Thus, the Agency was evaluating precision of the test for volatiles when the Federal Register proposal appeared on June 13, 1986. The final report on precision of the TCLP for volatiles was made available to the public on July 9, 1986 (51 FR 24856).

Ruggedness testing was conducted with the same industrial wastes used in the precision evaluation. The Agency concluded that the proposed test is adequately rugged. Evaluation of the procedure for volatiles was ongoing at the time of the June 13, 1986 proposal. The final report on ruggedness was made available to the public on July 9, 1986 (51 FR 24856). An Agency sponsored collaborative study (involving 26 laboratories, 5 wastes, and both volatile and non-volatile parameters) was also in progress at the time of the June 13, 1986 Federal Register proposal. The results of this study were made available to the public on September 19, 1986 (51 FR 33297).

The Agency received thirty-three comments which addressed various issues related to this research. The comments addressed the collaborative evaluation and the ruggedness and precision evaluations. All comments are summarized below.

### A. Collaborative Evaluation

The Agency received a total of sixteen comments which addressed the collaborative evaluation. One expressed support for the design of the study. However, most criticized the lack of complete information on the collaborative study available at the time of the June 13, 1986, Federal Register notice. Several also addressed the study coordinated by Lancy Environmental Services and funded by six trade associates (Lancy, 1986).

Several commenters were interested in including a particular type of waste (i.e. sewage sludge, fly ash, oily waste) in the study.

The Agency believes that it is unnecessary to perform precision studies on all wastes that may be subject to the TCLP since such an effort would be very costly in terms of time. In precision studies, it is more important to test a range of wastes (in terms of physical and chemical characteristics). The Agency also points out that the trade association study included many of the suggested wastes, and the results obtained were not significantly different from the results of EPA's study.

Twelve comments expressed a need for more information and urge the agency not to promulgate the TCLP until the results of the collaborative study have been made public and the regulated community has had a chance to comment. One comment stated that the proposed regulations are premature. It also stated that a thorough analysis of the comprehensive multi-laboratory evaluation results should have been conducted before the proposed rules were issued, rather than publishing the proposed rules with the provision that the collaborative study results would be available for analysis at a later date. According to the comments, the TCLP test simply has not had the benefit of appropriate peer review.

EPA notes that the results of the collaborative study (completed in September, 1986, and noted in the Federal Register at 51 FR 33297) indicate that the TCLP can be applied consistently by a diverse group of organizations. Total recoveries for both semi-volatile organic compounds and metals were sufficiently good to indicate that the variability falls within reasonable statistical limits (95 percent confidence level) for these two groups of constituents. The pesticide results were distorted by the limited amount of data and the wide range of detection limits reported by the participating laboratories. The Agency believes that a larger data set would yield results comparable with those derived for the semi-volatile organic compounds.

Nine comments referred to the study conducted by Lancy Environmental Services and funded by six trade associations and recommended that the Agency consider the results of this study prior to finalizing the TCLP.

EPA states that the results of the inter-industry association study (also available in September, 1986) indicate that the TCLP and EP toxicity tests are similar in precision. The relative standard deviation for both tests range from approximately 10% to 100% depending on waste type and parameters measured. The intra-laboratory precision was considerably better than the inter-laboratory precision. Both the lack of homogeneity in the wastes and the resulting difficulty in obtaining representative samples contributed significantly to the uncertainty surrounding the TCLP values.

Additional comments addressed the collaborative study report made available in September, 1986 (S-Cubed, 1986a). Several of these suggested that EPA should have included a comparison of the TCLP and the EP in the collaborative study. EPA decided that since both the Lancy study (Lancy, 1986) and a study conducted by the Electric Power Research Institute (EPRI, 1986) included a comparison of the TCLP with the EP, the Agency should concentrate its resources on the TCLP.

Other comments requested that EPA publish a final report on its collaborative study (the September report contained only preliminary results for volatiles) and make the final report available for public review and comment. One commenter recommended that the final report should contain the original data reports submitted by participating laboratories.

The Agency notes that a draft final report on the collaborative study has been prepared (S-Cubed, 1986b). Since the conclusions of the study were not changed in the final report, the Agency determined that it was not necessary to notice the report for comments again. Inclusion of the laboratory data reports would greatly increase the volume of the report without significantly adding to its content. These reports are available in the docket.

Some comments questioned the method used by the referee laboratory to identify outlier data points, and one participating laboratory noted discrepancies between its reported results and the data presented in the September report.

The Agency points out that the method used by the referee laboratory to determine outliers is described more fully in the draft final report (S-Cubed, 1986b). Data reported on compounds other than those identified as "target compounds" by the referee laboratory were not included in the report.

## B. Ruggedness Evaluation

Three comments criticized the ruggedness evaluation and express a need for additional testing. These comments asserted that the Agency failed to complete a ruggedness study of the TCLP at the time of the June 13, 1986, proposal. The comments also questioned the promulgation of the new test until the precision, accuracy, and ruggedness have been fully evaluated.

The Agency refers the commenters to results of the ruggedness evaluation described in the July 9, 1986, issue of the Federal Register (51 FR 24856), and the discussion of those results in the TCLP background document supporting the Land Disposal Restrictions Rule (US EPA, 1986b).

## C. Precision Evaluation

The Agency received fourteen comments which addressed the precision evaluation, and all were critical of the agency's procedures and results.

One trade association stated that the reported coefficients of variation for non-volatile components were quite high and that the precision of the test for low levels of non-volatile compounds was poor. The comment recommended that EPA reevaluate whether or not the precision of the TCLP test is adequate. Four comments stated a need for further testing and evaluation of method precision. They recommended that EPA vigorously pursue its methods activity on evaluation and development and then make its results available for public comment before final promulgation.

Two comments expressed concern about the lack of data on the precision of the test for volatiles at the time of the June 13, 1986, proposal. They urged EPA to ensure the development of adequate precision and accuracy data for volatile organics. One comment questioned whether disposal companies will be unwilling to rely on such a highly variable test for assurance of a waste being nonhazardous.

EPA notes that it conducted single and multi-laboratory evaluations of the precision of the TCLP for volatiles and non-volatiles, as did two industry groups (EPRI and the trade association study coordinated by Lancy Environmental Services). These efforts support the adequacy of the precision of the TCLP and also support EPA's contention that precision over the existing EP has been improved, if only slightly. Specifically, these studies show that both sampling and analytical variability contribute significantly to the total variability of the TCLP. (S-Cubed, 1986a; S-Cubed, 1986b; Lancy, 1986; EPRI, 1986).

EPA refers the commenters to Table 3 which shows the mean and median relative standard deviations for the major classes of analytes (averaged over the various laboratories, samples, and individual analytes for each of the three collaborative studies). It should be noted that the average or median percent relative standard deviation for each class includes the variability attributed to different waste streams, different samples of the same waste stream, different laboratories, the leaching procedure, different analytes, and the analytical procedures. Although a quantitative analysis of variance has not been performed on these data, the collaborative studies indicate that the largest contributors to this variability are the differences between samples of the same waste. The variability of the TCLP is likely to be as significant as the analytical method variability. However, it is reasonable to assume that, since the analytical method variabilities are considered acceptable, the TCLP variability is acceptable. The focus on reducing variability should be placed on sampling procedures as well as the leaching test and subsequent analyses since they contribute significantly to total variability.

Some comments expressed concern about poor reproducibility of the test for metals. One comment referred to a study conducted by a trade association which showed the metal concentration to be very sensitive to sample preservation, particle size distribution, and variations in lab procedures. Another comment asserted that the test is inadequate because they observed a relative standard deviation greater than 100% in the results of tests performed by contract laboratories. A utility company asserted that the precision of the TCLP should be evaluated for all 8 EP metals, since performance varies with the analyte.

The Agency responds that, as demonstrated by all three collaborative studies mentioned previously, precision of the TCLP for metals is within the realm of acceptability. In particular, the EPRI study concluded that the precision of the TCLP is at least as good as that of the existing EP test.

A minerals processing firm believed that the precision data are misleading in that the degree of precision is primarily due to the aggressiveness of the leach liquor.

## TABLE 3. RESULTS OF TCLP COLLABORATIVE STUDIES
## % RELATIVE STANDARD DEVIATION

| % RSD | EPA | IIA | EPRI |
|---|---|---|---|
| Chlorinated Volatiles: | | | |
| Mean | 77 | 54 | - |
| Median | 76 | 42 | |
| Range | 17-127 | 24-78 | |
| Non-Chlorinated Volatiles: | | | |
| Mean | 71 | - | - |
| Median | 69 | | |
| Range | 27-144 | | |
| Semi-Volatiles: | | | |
| Mean | 85 | | |
| Median | 81 | - | - |
| Range | 0-164 | | |
| Pesticides: | | | |
| Mean | 160 | | |
| Median | 160 | - | - |
| Range | 7-278 | | |
| Metals: | | | |
| Mean | 67 | 31 | 49 |
| Median | 67 | 21 | 52 |
| Range | 3-129 | 7-63 | 9-104 |

EPA: multi-laboratory collaborative study coordinated by S-Cubed
IIA: inter-industry association study coordinated by Lancy Environmental Services
EPRI: Electric Power Research Institute Study of utility wastes

The Agency agrees that the more aggressive the leaching media, the more reproducible the results (for metals). One need only consult the results of acid digestions to confirm this.

An oil company stated that EPA needs to evaluate the precision and ruggedness of the TCLP for multiphase samples.

The Agency points out that one of the wastes used for evaluation of precision for volatile analytes was a mixture of API separator sludge and electroplating waste (S-Cubed, 1986d). Studies conducted using oily wastes led to the Agency's decision to specify a glass fiber filter for the TCLP rather than the membrane filter used in the EP based on improved filtration and reproducibility for the oily wastes studied (ERCO, 1985). Further studies are underway to investigate whether changes to the TCLP can enhance the tests ability to model the behavior of oily wastes in a land disposal environment.

A chemical company asserted that too much emphasis has been placed on precision of the test since sampling of waste streams is not a very precise operation. The company believes that the procedure can be simplified and made less expensive with no loss of data value.

EPA responds that it has made a serious, conscious effort to produce the simplest procedure possible. As indicated in other sections of the preamble and background document, EPA is also investigating further ways to simplify the method (principally the ZHE portion) and thus may propose more changes. The Agency agrees that sampling contributes significantly to the overall variability of the procedure, as shown by some of the collaborative studies. Attention must be paid to reducing all sources of variability in the procedure.

## IV. General Leaching Procedure Issues

Various aspects of the procedure were reviewed by EPA and others, and a number of topics were identified as Leaching Procedure Issues. These issues include: (1) the accuracy of the TCLP; (2) the procedure for moderate to highly alkaline wastes; and (3) the pre-screening test. Seventy-three comments addressed these issues. The comments were made by trade associations, the petrochemical industry, mining and mineral companies, waste management firms, other private industries, municipalities, other government agencies, environmental consulting firms, and private utility firms and research laboratories. A detailed discussion of each issue follows.

### A. Accuracy of the TCLP

EPA was directed by the HSWA to make the EP more accurate. The experimental program for developing the TCLP was intended to provide an accurate model of a field co-disposal situation. One of the leaching procedure issues is whether the method is adequate in this respect. Twenty-four comments criticized the accuracy of the method.

Most of these comments recommended additional testing to improve the level of accuracy thus far demonstrated by the TCLP. These comments noted that, in the development of the TCLP, only eleven wastes were tested. Increasing the variety of wastes and the number of extractions performed may result in method refinements that, in turn, will enhance the accuracy. Several specific types of wastes were suggested for additional testing, including oily waste, organic chemical wastes, and municipal wastes. Two comments questioned the Agency's basic definition of accuracy and its accompanying documentation described in the proposed rules. Five comments stated that they do not believe that the 182% relative standard deviation reported in the proposed regulation is a sufficient level of accuracy for meeting mandated requirements of the HWSA. One comment suggested that the procedure be repeated several times on each waste stream in order to improve the accuracy of determining the hazardous constituents over a period of time.

The Agency believes that the accuracy of the TCLP is adequate in terms of the test's ability to indicate the potential for wastes to pose a hazard if mismanaged. The laboratory test system reproduces the field lysimeter leachate concentrations for about three-fourths of the target compounds within one standard deviation of the distribution. The field-scale lysimeters are the most practical method available for simulating the behavior of actual landfills. The Agency also believes that the eleven wastes tested during the method development process adequately represent the range of waste types encountered in the real world (with the exception of alkaline wastes, which are discussed below).

Several comments suggested that the TCLP does not accurately measure the hazard posed by oily wastes because it assumes that liquid wastes (both aqueous and non-aqueous) behave in an identical fashion in the subsurface environment. The comment also contended that the Agency provides no evidence to support this assumption and therefore urges the Agency to conduct further investigations into the behavior of oily wastes.

The Agency points out that the TCLP is designed to predict the release of contaminants from landfilled wastes into the subsurface environment. The mobility of the released aqueous or non-aqueous phases through the saturated and unsaturated zones of the subsurface is addressed by ground water modeling. Three of the eleven wastes investigated during TCLP development represented oily wastes. This research demonstrated that non-aqueous liquids are released from landfilled wastes. Such release is modeled by the liquid/solid separation step of the TCLP. Additional investigations are underway to evaluate whether modifications the the filtration step can more accurately predict the release of aqueous and non-aqueous liquids into the subsurface.

## B. Treatment of Highly and Moderately Alkaline Wastes

EPA is concerned that the potential hazard posed by alkaline wastes may be underestimated by the acetate buffer system initially chosen for the TCLP. Specifically, EPA believes that an increase in the leaching of inorganic and some organic species may be observed as the alkalinity of the wastes becomes exhausted (due to continuous contact with an acidic leaching medium). The TCLP acetate buffer leaching fluid may therefore not adequately account for the leaching of heavy metals from wastes of moderate to high alkalinity. Thus, EPA proposed a two leaching fluid system for the TCLP. The Agency chose to base the strength of the alkaline waste leaching medium on 2 milliequivalents of acid per gram of waste (the maximum amount of acid allowed in the EP) for wastes of moderate to high alkalinity and 0.7 milliequivalents per gram of waste for other wastes. This dual leaching fluid system is not proposed for evaluating volatile compounds since these compounds are expected to be unaffected by slight changes in acidity.

Eighteen comments were concerned with the application of the TCLP to alkaline wastes. Six of these comments noted that no highly alkaline waste was included in the development of the TCLP and that no conclusions therefore can be made concerning the actual behavior of these wastes. Four comments stated that the leaching medium in the municipal landfill is relatively constant regardless of the type of industrial waste co-disposed. Therefore, there should be a single leaching medium for the TCLP that applies to all types of wastes. Six comments contended that the more aggressive leaching medium used for the TCLP is not justified because it is much more acidic than the actual leachate examined in the ORNL Phase II study and in the collected data on municipal landfill leachate.

Three comments expressed concern about the inaccuracy that might appear in subsequent analyses of alkaline wastes as a result of the more acidic leaching medium. One comment stated that the procedure used to determine which leaching fluid should be employed may result in very different results from tests performed on very similar wastes. Based on the alkalinity test, two wastes may be subjected to different leaching media, when the wastes actually behave very similarly in the field. A mining and metal industry comment expressed concern that use of acetic acid as the leaching medium would falsely exaggerate the potential availability of metals for migration. A municipal sewer district feared that lime-stabilized sewage sludge might be reclassified as hazardous when tested with the TCLP procedure for alkaline wastes. The comment maintains that according to actual disposal practices, sewage sludge is never exposed to acidic leaching conditions.

The Agency acknowledges the fact that highly alkaline wastes were not included in the TCLP development program. For this reason, EPA is concerned that the acetate buffer leaching medium may underestimate the potential hazard posed by such wastes. Therefore, a leaching medium containing 2 milliequivalents of acid per gram of waste (the maximum amount of acid allowed by the EP) is required for wastes of moderate to high alkalinity. A leaching medium containing 0.7 milliequivalents of acid per gram of waste is required for all other wastes and for all analyses where volatiles are involved.

## C. Pre-screen Test

The pre-screen test consists of a total waste analysis using SW-846 methods and determines if the waste contains enough specific compounds to exceed the regulatory level. This test assumes that all of the compound leaches from the waste. If, based on such an analysis, one can be certain that the regulatory level cannot be exceeded, the TCLP does not need to be performed. Thirty-one comments addressed the pre-screen procedure. Nineteen comments totally supported the Agency's proposed procedure for providing a method of avoiding excessive analytical cost and time.

Several comments requested additional guidance from the Agency regarding performance of the pre-screen test, and one comment suggested that the pre-screen test be incorporated as a provision of 40 CFR 261.24.

The Agency does not believe that it is necessary for the method to include a step-by-step description of the pre-screen test. The pre-screen test directs generators to: conduct total waste analyses using SW-846; assume that all the constituents, if present in the waste, migrate into the leachate; and determine whether the constituent has the potential to exceed the appropriate regulatory level. Selecting the most appropriate SW-846 method for a total analysis is a waste-specific determination. Thus, it is best performed by the laboratory performing the analyses. The calculation of maximum extract concentration is easy for non-liquid wastes. The waste concentration (mg/kg) is merely divided by a factor of 20 to predict the maximum possible TCLP extract concentration. The calculation is only slightly more difficult for wastes containing liquids. This calculation requires separate analytical determinations of the relative constituent loadings of both liquid and solid phases and a simple volume weighted average of the concentration of the constituent in the liquid phase and the concentration in the solid phase (mg/kg) divided by a factor of 20. EPA believes that these calculations are straightforward enough that they are not spelled out in the method.

Other comments offered general support for the pre-screen procedure but make suggestions for improvements. Three comments suggested that, in addition to a pre-screen test, the generator's knowledge and experience should be used in deciding what constituents in the waste require analysis. Another comment advocated the establishment of a recognized method whereby a generator can avoid the expense of routine analyses of constituents known to be absent from a waste stream.

The Agency responds that process knowledge and knowledge of the waste itself is allowed under the Toxicity Characteristic (40 CFR 262.11) for determining whether wastes are hazardous as defined by the hazardous waste characteristics.

One comment suggested that it is unnecessary to analyze the total waste if a single phase of the waste contains such a high concentration of a constituent that the regulatory threshold would be exceeded regardless of the concentration in the other phases or extracts. Another comment suggested that volatiles be measured in the aqueous phase of the waste because the leachate derived from the TCLP probably would not contain greater concentrations of volatiles than in the aqueous phase of the waste. The comment stated that this could be especially applicable for oily waste.

EPA generally agrees. However, this may be true only when the waste contains a small "solid" phase (e.g., less than 10%). EPA has no data that would support this contention.

Several comments criticized the Agency's proposed pre-screen test. One comment stated that the cost of performing the pre-screen test will exceed the cost of performing the TCLP.

EPA agrees that the cost of the total analysis may indeed equal or even exceed the cost of performing the TCLP and running subsequent SW-846 analyses. However, many generators have the results of total constituent analyses of their wastes already available which can be used in this evaluation.

Another comment questioned whether commercial waste disposal firms will accept the results of a pre-screen test in lieu of a complete TCLP.

EPA responds that the pre-screen test provides generators with the means by which to demonstrate that their wastes either do not contain certain constituents, or that if they do contain these constituents, to predict whether the maximum possible TCLP extract concentration falls above or below the appropriate regulatory level. The pre-screen test is a part of the method and its results must be accepted as equivalent to the results of the leaching procedure itself.

## V. Specific Technical Comments on the TCLP

One half of all the comments which the Agency received addressed specific aspects of the TCLP. The areas of comment included the extraction vessels (ZHE), the agitation apparatus, the borosilicate glass fiber filter, the leaching media and other reagents, the particle size reduction requirements, specific steps in the extraction procedure, the quality assurance requirements, and the relationship between the TCLP and the leaching tests used in the Agency's delisting program.

### A. Extraction Vessels (ZHE)

Fifty-nine comments expressed concern regarding the extraction vessels specified. Five comments voiced qualified support for the use of the ZHE for volatile compounds and fifty-four comments criticized the use of the ZHE for volatiles.

Several comments expressing qualified support for the ZHE suggested design improvements to further minimize loss of volatiles. These comments recommended that the internal volume of the ZHE be reduced to approximately 110 percent of the volume of the waste charged into the ZHE in order to further minimize the headspace and loss of volatiles. Another comment stated the ZHE is of high quality design even though it is expensive.

The Agency agrees that the ZHE design is of high quality and that the internal volume of the ZHE should be reduced in order to minimize the headspace. Therefore, the method has been changed to clarify that, prior to charging the ZHE with sample, the piston should be moved to a position which will minimize both headspace and the distance the piston must move to express the initial liquid phase of the waste.

Thirteen comments critical of the use of the ZHE questioned the availability of the apparatus and are concerned about the manufacturers' ability to meet demand. A typical comment stated that the commercial availability should be assured prior to the effective date of the regulation.

EPA replies that the ZHE is now available through additional manufacturers. One manufacturer reports almost a full year's inventory currently in stock. Also, the accessories needed for the TCLP (i.e., glass fiber filters, Tedlar bags, syringes, etc.) are now readily available.

One of the comments addressing the availability of the ZHE pointed out that the manufacturers experienced some serious problems during the initial testing of the ZHE.

In response, the Agency explains that one initial problem was a loss of volatiles through pressure and liquid release valves. This problem has been rectified through changes in the TCLP (including a sequence of steps that will allow the equipment to be checked for leaks). See Step 4.2.1 for further information.

Many of the comments which criticized the ZHE addressed the inconsistencies between the ZHE and extraction bottles. Several of these comments requested data indicating the necessity for a device like the ZHE. For example, one comment asserted that EPA has not justified the need for more complicated and more expensive laboratory equipment such as the zero-headspace extraction vessel.

In response, the Agency refers the reader to the S-Cubed report (S-Cubed, 1986c) on single laboratory testing using the conventional apparatus. The report concludes that the conventional TCLP extraction vessel (bottle) is not feasible for extraction of volatile compounds. Recoveries were usually less than 25% for spiked samples. The losses were attributed to the headspace present in the bottles during extraction and the vacuum filtration used for liquid/solid separation. These results led to the development of the ZHE.

One comment addressing the inconsistencies between the ZHE and the bottles supported the use of a standardized method of extraction for volatiles and non-volatiles in order to obtain comparable results. Another comment revealed that different analytical results for non-volatile and inorganic constituents were obtained using the two devices and suggests that the difference occurs because the agitation action in the ZHE differs from that of the extraction bottles. Other comments questioned the size of the ZHE (specified as 500 milliliters) when it is known that the non-volatile sample size of 100 grams will require a volume of more than 2000 milliliters.

EPA states that the ZHE is not intended for analysis of non-volatile compounds. The wording in the procedure has been modified to emphasize that the ZHE is to be used only for volatile analysis (See discussion in the March 10, 1986, TCLP Background Document (USEPA, 1986a)).

Another area of concern was the corrosion of the stainless steel walls of the ZHE. This corrosion could cause contamination of the leachate, producing unrepresentative results. Many comments which are critical of the use of the ZHE expressed concern in this area. One comment asserted that the use of a stainless steel vessel for extraction of inorganic materials should be avoided since the nature of the wastes, when combined with the potentially abrasive action between the waste and the container walls, may contaminate the leachate with elements from the steel. Another comment stated that the stainless steel could corrode, producing high blank levels and unrepresentative results.

The Agency points out that the ZHE is only intended for analyzing volatiles, thus potential metals contamination is not a concern.

One comment remarked that, if the ZHE is required, then the borosilicate glass bottle should also be specified for the metals analysis.

EPA agrees. The TCLP has been modified to recommend the use of borosilicate glass bottles over the use of flint glass bottles when analyzing for inorganics. Flint glass may cause interferences in the inorganic analysis. However, flint glass can be used in the analysis of non-volatile organics. Also, "plastic" bottles can be used if only inorganics are to be investigated.

Six comments which are critical of the use of the ZHE stated that the specified volume of 500 ml for the ZHE is not adequate to contain the sample size required for extraction. Several of these comments suggested that a problem exists with the ZHE for waste streams containing 0.5% (or greater) solids. For example, one comment stated that, in order to obtain a 25 gram solid sample for extraction, the filtering process would produce about five liters of filtrate. The capacity of the ZHE is only 500 ml. Therefore, the contents would be exposed to the atmosphere up to ten times during the procedure. As a result, one comment asked if the ZHE is of any benefit if it is opened and closed too often when trying to meet the sample size criterion.

Regarding use of the ZHE with a waste stream containing low percent solids, the Agency points out that the TCLP specifies only a maximum (25 grams of solid) and not a minimum sample requirement for the volatile procedure. The proposed method recommended that the ZHE be charged only once. The method has now been modified to state clearly that the ZHE should only be charged once and that there is no minimum sample size requirement as long as enough leachate is generated to perform the necessary analyses. If insufficient leachate is generated by a single extraction, multiple extractions may be performed and the leachate combined for analysis.

One comment suggested that for maximum pressure of 50 psi, the ZHE could be made with less metal. This would make it possible to have a larger vessel and a bigger sample size.

In response, the EPA asserts that extraction with the ZHE may involve pressures greater than 50 psi. For safety reasons, the walls of the vessel should not be made thinner. Moreover, the Agency believes that the specified sample size is more than adequate for analyzing volatile compounds.

Several comments expressed concern over the difficulty they experienced in cleaning the ZHE. For example, one comment was concerned that the Viton "o" rings absorb organic compounds, regardless of the attempts to clean the material.

The Agency points out that the Viton "o" rings should be replaced frequently, as stated in step 4.2.1 of the TCLP method, thereby reducing the risk of contamination.

Another comment described the difficulty encountered while cleaning syringes and Tedlar bags for re-use. This comment continued by urging the EPA to consider VOA vials, which are less expensive, portable, durable, and disposable or easily cleaned, for collection of the TCLP extract.

The EPA states that Tedlar bags are not re-usable. VOA vials can be used to store extraction fluid. However, they are not recommended for direct collection of the filtrate because there is no way to connect the VOA vial with the ZHE in such a manner as to exclude headspace.

One comment suggested improving the ZHE to allow ease of cleaning. Specifically, this comment recommended that the spacing of the support screen for the filter be made only as small as absolutely necessary.

The Agency states that the specified size of support screens are needed to protect the GFF filter from abrasion by the waste.

A few of the comments stated that sometimes it is impossible to clean the ZHE and that it has to be discarded. As a remedy to the situation, one comment urged the use of a disposable liner.

In response to these comments, the Agency explains that the concept of disposable liners has been considered but was rejected as impractical. One reason is that an exact fit is required for the piston inside the ZHE barrel, and the use of a liner will not allow a secure fit. Also, the Agency is currently investigating cleaning procedures to be included in the method. If the ZHE is found to be impossible to clean, then the manufacturer should be contacted for inspection of the device.

Some of the comments concerned with the extraction vessels expressed an interest in safety precautions with respect to pressure build-up. One comment asserted that EPA should evaluate the safety aspects relative to wastes which could react and produce off-gases. Specifically, they were concerned with possible pressure build-up inside the ZHE from reactions of highly alkaline wastes. A few of the comments suggested that the ZHE should be equipped with a device for releasing pressure. One comment stated that a pressure release valve on the ZHE might remedy the situation. However, another comment suggested that venting of gases due to pressure build-up may result in the loss of volatiles.

Recognizing the possibility of pressure build-up, EPA states that the stainless steel containers have been designed to accommodate high pressures. The ZHE is also equipped with pressure release valves. However, when using extraction bottles, EPA recommends venting the bottles shortly after the waste contacts the extraction fluid. Regarding the loss of volatiles during venting, the EPA points out that it is not possible to completely avoid a loss of volatiles although the TCLP is designed to minimize losses.

Several comments offered possible alternatives to the ZHE. Specifically, these comments asserted that comparable results may be obtained with a standard 40 ml glass VOA vial with a Teflon-lined septum for sample extractions. They recommend that the Agency determine whether or not a simpler system would yield comparable results for the volatile organics. Additional comments suggested that a screw-top bottle with a suitable cap liner (filled full) is an adequate zero-headspace device. One comment requested that the Agency provide data demonstrating that the ZHE produces more representative results than other existing devices which are more readily available and easier to use.

EPA responds that it is investigating the option of a simpler extraction system. However, the 40 ml VOA vial is not a suitable alternative. A 40 ml VOA vial would not permit solid-liquid separation to be performed under minimum headspace for the specified sample size. EPA's studies of precision for volatile compounds using both the ZHE and the conventional apparatus indicate that the device does reduce losses. (S-Cubed, 1986c; S-Cubed, 1986d)

## B. Agitation Apparatus

Thirteen comments criticized the rotary agitation apparatus. The areas of concern include the effects of rotation on particle size, the use of the rotation apparatus with the ZHE, the rate of agitation, and the agitation time requirement.

Several of these comments stated that the rotary apparatus will cause particle size reduction, resulting in non-representative extractions of constituents. One comment stated that the rotary cells used in the procedure may cause mechanical grinding of monolithic wastes. Another comment stated that the agitation apparatus will produce unrepresentative high concentrations of contaminants (based on the chipping that will occur during agitation). Another comment stated that the TCLP is inappropriate for vitreous and other hard waste forms since the tumbling process grinds the waste into finer and finer particles. Also, the cement and lime based waste forms may paste up during the TCLP, thus limiting the exposure of the waste to the leachate. Therefore, this comment questioned whether or not the TCLP ensures a complete and effective contact between the waste sample and the leachant.

The Agency replies that the extraction is intended to be representative of leaching over a long period of time. In order to simulate this long-term leaching, batch leaching tests with agitation are required to ensure that equilibrium between the extractant and the sample is achieved within eighteen hours. For additional information, the Agency refers to the Background Document prepared for the EP test (US EPA, 198C).

Some comments which addressed the use of the rotary agitation apparatus disapproved of the its use with the ZHE. For example, a few of the comments pointed out that the rotary apparatus and the extraction vessel must be purchased from the same vender in order to assure compatability.

In response, EPA asserts that most agitation apparatuses and extraction vessels are compatible with minor adjustments. Therefore, the devices may be purchased from different venders.

Another comment suggested that the use of stainless steel balls in the ZHE would increase and improve mixing. Thus, the extractor would simulate a ball mill.

The Agency has no data indicating that mixing is a problem in the ZHE. The Agency has evaluated the use of stainless steel balls for particle size reduction, not necessarily for mixing. However, the steel balls resulted in damage to the ZHE.

Many of the comments which were critical of the rotary agitator would like the EPA to re-consider the agitation rate of 30 +/- 2 RPM. Several comments questioned whether or not using the rotary-tumbler at a fixed agitation rate of 30 +/- 2 RPM simulates actual landfill conditions. One comment criticized the continuous agitation action, stating that continuous agitation during extraction is an unnecessarily conservative condition. Another comment recommended that the EPA reconsider the rotary tumbler for some wastes (i.e., dewatered sludge). This comment asserted that more realistic results will be produced if vigorous shaking is employed.

The Agency explains that continuous agitation is necessary in order to ensure equilibrium between the extractant and the sample. Uniformity in mixing of the sample and extractant is an important factor in method precision. The rotary tumbler apparatus is one of the most readily available and least expensive methods of achieving the goal of effective mixing. Alternatives were considered, including the use of stirrers as allowed in the EP, but they were determined to be more costly and/or error prone. The agitation rate of 30 +/- 2 RPM ensures complete contact between the sample and the leaching medium.

## C. Filters

Twenty five comments addressed the use of the borosilicate glass fiber filter which has a pore size of 0.6-0.8 µm. Only one of these comments provided qualified support for the filter. The remaining comments were negative.

One comment believed that the filtering method has been improved. However, the protocol should provide for changes of the glass fiber filter since the filter tends to become plugged (even with careful application of gas pressure).

The Agency agrees and states that, for the final filtration in the TCLP (non-ZHE), the filters can be replaced. However, the ZHE cannot be opened to replace filters. The Agency has modified the wording of the TCLP to reiterate that centrifugation may be used as an aid for filtration in the non-volatiles procedure.

One comment expressed strong reservations about the EPA decision to change the filter size in the TCLP. Specifically, it requested that the EPA explain the basis for this change. If the filter is retained in the final rule, EPA should explain how the change in filter size will improve testing accuracy.

The Agency refers to the March 10, 1986, TCLP Background Document (US EPA, 1986a) as the basis for using the borosilicate glass fiber filter. The decision to change filter types was based on operational reasons: the glass fiber filters do not clog as easily as the 0.45 µm membrane filters, and the Agency believes that the glass fiber filters will provide a more adequate differentiation between those materials which behave as liquids in the environment and those which behave as solids in the subsurface environment.

The remaining comments addressed four specific areas of concern: filtering oily waste; loss of integrity of filter when wet; particulates in the filtrate; and the availability of the specified filter.

Many comments expressed concern regarding the filtering of oily waste. Several of these comments discussed the effect of dissolved and suspended oil in the extract. One comment claimed that dissolved and suspended oil will be recovered in the filtrate when filtering oily waste. This will yield a highly biased recovery of components dissolved in the original oil, which does not reflect the true mobility of these components. Another comment re-emphasized the observation that the increased pore size of the filter allows oil to pass through and into the filtrate.

The Agency is investigating waste-specific modifications to the TCLP. If oily material passes freely through the filter, then it will be mobile and can migrate to the ground water. In fact, dissolved and suspended oil has been found in ground water in many areas. Therefore, there is no reason to exclude oily liquids from the extract.

One comment stated that oily wastes plugged the filter within the first 15-20 seconds of the filtration process. Another comment addressed the problems associated with filtering water immiscible organics and other wastes. Specifically, it stated that, even with a larger pore size, the immiscible organic material will coat the filter fibers and produce a hydrophobic surface that either impedes or completely stops the filtration.

The Agency states that, if the method is followed with incremental increases in pressure, the plugging problem will be minimized. To help the process of filtration, centrifugation may be used to facilitate the initial liquid/solid separation when the non-volatile procedure is used. However, if liquid remains in the filtration unit, then for now it should be treated as a solid. The Agency acknowledges that, when filtering difficult samples, problems will always exist. Waste-specific modifications to the procedure are under investigation.

Another area of comment was the loss of integrity of the filter when it is wet. Two comments criticized the filter on these grounds. According to one comment, experience has shown that the loss of integrity makes it impractical to wash the filters prior to use. The TCLP should specify that the filters be washed after the filtration device has been assembled.

The Agency states that the recommended filters, if handled carefully, will not lose their integrity when wet. Acid washing after the filtration device has been assembled is not recommended. The Agency believes that it is difficult to remove all the acid from the filters and, as a result, may alter extract concentrations (i. e., metal concentration).

Several other critical comments addressed the problem of particulates passing through the filter into the filtrate. According to these comments, the glass fiber filter is likely to allow many more large particulates to pass through the filter than the filter which was used in the EP did. One comment suggested that the TCLP should allow for the clarification or removal of any fine particulates which might pass through the less selective glass fiber filters, and asserts that the TCLP should not include the burdensome acid digestion for metals analysis.

EPA believes that switching from the membrane filter to the glass fiber filter will not increase significantly the passage of particulates into the extract, and that particles which pass through the filter will also be mobile in the subsurface environment. The Agency notes that acid digestion has always been required for the EP, and will continue to be required for the TCLP.

One comment stated that the EPRI-commissioned "round-robin" study shows that metal contamination, in conjunction with the glass fiber filter, is high (even when the filter is washed with acid as described in the TCLP proposal). This comment recommended washing the glass fiber filter with at least 1.5 liters of the acid solution, to be followed by rinsing with two liters of de-ionized distilled water. The commenter believed that this rinsing will provide a satisfactory means of removing contamination in the glass fiber filter.

The Agency states that the EPRI study revealed that only one laboratory showed a problem while using the glass fiber filters and this was traced to contaminated dilution water (EPRI, 1986). Also, step 4.4 of the TCLP method provides an adequate acid washing procedure.

Three comments objecting to the use of the specified filter addressed the unavailability of the necessary size of glass fiber filter. One comment stated that the specified size of the filter holder is 142 mm. However, the only acceptable filter is the Whatman GFF which is not available in the 142 mm size.

EPA acknowledges the fact that the Whatman 142 mm glass fiber filters are not readily available; however, Whatman's 150 mm filters can be readily adapted for use.

## D. Reagents

Four comments addressed reagents used in the TCLP. The areas of interest included the daily preparation of the extraction fluid, reagent grade glacial acetic acid, adjusting the buffering solutions, and storage for ASTM type 1 water.

Three comments addressed the requirement that the extraction fluid be prepared on a daily basis. One comment questioned why the extraction fluid must be prepared daily if the pH of each of the extraction fluids is to be evaluated before use and if there is one blank for every 10 extractions. Furthermore, if the pH of the leaching fluids meet the criteria and the method blanks are free of contamination, then it may be more cost effective to prepare a large supply of the leaching fluid rather than preparing a small volume on a daily basis. Another comment stated that the Agency has presented no rationale for the requirement which states that the leaching reagents must be prepared on a daily basis. This comment also stated that the proposed requirement imposes unnecessarily harsh conditions on the laboratories.

The Agency notes that the daily preparation of the extraction fluid is now suggested rather than required. (See Note at 5.6.2.) Experience has shown that short term deterioration of the extraction fluid is not a problem.

One comment responded specifically to the wording contained in step 5.5. It pointed out that the phrase "made from" in step 5.5 should be replaced with a semicolon or a colon. This change will clarify the fact that glacial acetic acid may be purchased as a reagent grade. It is not prepared that way in the laboratory.

The Agency agrees and the TCLP method modifications have changed the wording of step 5.5 to read glacial acetic acid is ACS grade and not prepared from ACS grade.

One comment expressed some confusion regarding the preparation of reagents to be used in the TCLP and recommended that specific directions be given for the adjustment of buffering solutions. It also suggested that the ASTM Type 1 water used in the analysis of volatiles be stored under inert gas or with no headspace to minimize contamination.

In response, the Agency states that specific directions are not necessary since buffering solutions are not adjusted. If the buffering solutions are prepared according to the directions, adjustment will not be necessary. The Agency refers to the TCLP method changes and states that the TCLP will require Reagent Water instead of ASTM Type 1 water. (See Step 5.1.)

## E. Leaching Media and Liquid/Solid Ratio

Thirty-four comments criticized the dual leaching media composition. One comment expressed concern over the effects of the sodium acetate buffer on specific wastes. For example, the use of the buffered extraction fluid in the TCLP appears to be designed for the extraction of volatile compounds in the ZHE device. Since alkaline wastes will not be neutralized by the extraction fluid or adjusted to a specific pH, the leaching of wastes for extractable organics and metals could be compromised. The use of distilled or ASTM Type 1 deionized water should be given consideration so that interferences, artifacts, and possible contamination could be minimized.

In response, EPA states that the buffered solution was chosen from ORNL research on both organic and inorganic wastes using the conventional bottle extractor. A more acidic leaching solution is required for alkaline wastes. Research has shown that leaching medium composition is more important for the leaching of metals than for the leaching of organics (Warner et al., 1981). EPA is investigating the use of distilled water for the extraction of volatiles.

Eleven comments addressed the effects of the acetic acid solution on alkaline wastes, natural buffering wastes, and mining wastes. Specifically, one comment stated that EPA's assertion that the TCLP and EP values should be approximately the same for sludges is not true for some weak to moderate alkaline wastes. Therefore, the TCLP should be modified to avoid undue over-regulating of EP nonhazardous wastes with weak to moderate alkalinity.

Several comments were concerned that the acetic acid solution is overly aggressive and will result in more wastes being regulated as hazardous. Specifically, one comment stated that the extraction fluid #2 is equivalent to the maximum amount of acid added under the current EP method. The criteria for selecting the extraction fluid will increase the number of wastes subject to the extraction procedure with the maximum amount of acid. Similarly, those wastes requiring very little acid to maintain pH 5.0 by the EP will be subject to extraction under more acidic conditions with extraction fluid #1 by the TCLP.

One comment expressed concern over alkaline or chemically-treated wastes. This comment stated that the stronger acid leach media will tend to destroy the stability of alkaline or chemically-treated waste. Another comment discussed the destabilizing effects of acetic acid on buffering wastes. This comment stated that the acetic acid solution penalizes wastes that have natural buffering capacity.

Several comments were concerned that the acetic acid solution will result in an unrepresentative and excessively high concentration of metals. One comment pointed out that acetic acid causes a greater amount of leaching of metals from a solidified mass than what occurs in nature. Acetic acid is not commonly found in nature in the concentrations used either in the EP toxicity test or the new TCLP test. Another comment stated that an acetic acid solution will overestimate (by up to several orders of magnitude) the amount of metals which could leach from the waste. Similarly, one comment stated that the change in extraction fluid could have a major impact on the solubility of metals during the TCLP. Therefore, a single extraction fluid (#1) should be applied to all wastes, regardless of pH, to avoid complicating steps and to establish a common set of operating conditions.

Two comments addressed the use of an acetic acid solution with mining wastes. These comments provided extensive discussions regarding the impact of using an acetic acid-acetate solution for mining wastes. The comments argued that these wastes are placed in monofill disposal sites and are not exposed to organic acids, which are extremely aggressive in solubilizing certain metals.

The Agency points out that the Oak Ridge studies show the acetate buffer leaching medium produces contaminant concentrations comparable to those observed in the field lysimeter leachate (Francis et al., 1984; Francis and Maskarinec, 1986). Comparisons of the TCLP with the EP conducted by the Electric Power Research Institute (EPRI, 1986) and a group of trade associations (Lancy, 1986) indicate no significant difference in the metals concentrations obtained using the two tests. The Agency recognizes that the TCLP was not designed for site-specific evaluations and is currently investigating other approaches to testing, including alternatives for regulating monofill disposal.

Several comments questioned the validity of the 20:1 extraction ratio. One comment requested that the EPA justify the selection of the 20:1 extraction ratio. Another comment pointed out that the ratio of 20:1 does not yield a concentration representative of an infinitely long extraction process. This comment also stated that the literature indicates that ratios closer to 10:1 in batch extractions represent more accurately the equilibrium values found in continuous flow column extractions. Therefore, the 20:1 ratio underestimates the concentration in the initial pulse of leachate from the site but overestimates the long-term equilibrium release. Another comment also questioned whether or not the 20:1 extraction ratio actually represents the effect of leaching in the disposal environment.

The Agency responds that the 20:1 ratio of leaching fluid to waste represents a reasonable compromise to approximate the average maximum exposure concentration. Since this regulation is primarily concerned with the chronic toxicity of compounds over medium to long exposure intervals, the 20:1 ratio is more appropriate than a lower ratio such as 10:1 or 4:1. The Agency acknowledges that the 20:1 ratio is based on practical as well as theoretical considerations. Research shows that a liquid to solid ratio less than 20:1 gives results that are less reproducible, while higher ratios present practical problems because of the volume of material to be handled. (Kimmel and Friedman, 1984).

Some of the comments which criticized the dual leaching media composition expressed concern regarding the pH requirement of the extracting fluid. One comment pointed out that the leaching media create acidic conditions which are much more exaggerated than those found in even the most conservative municipal landfill mismanagement scenario. Another comment recommended a neutral medium as an alternative to the acidic leaching medium.

The Agency repeats that the acetate buffer leaching fluid adequately reproduced the contaminant concentration obtained from the field lysimeters, as shown in the Oak Ridge research (US EPA, 1986a; Francis et al., 1984). The acetic acid leaching fluid contains the maximum amount of acid allowed for the EP test. Both natural rainwater and municipal landfill leachate tend to be acidic rather than neutral.

Two comments questioned the validity of using two leaching media. One comment asked if the TCLP is intended to represent a worst-case co-disposal scenario, what is the need for two different extracting solutions for wastes with different pH values. This comment stated that the pH of the leachate will not become more acidic if the landfill contains a basic waste. Several other comments also supported the use of a single extraction fluid. One stated that, if the EPA is predicting that a waste will be contacted by 200 milliequivalents of acid per 100 grams of sample, then that amount of acid should be used in all cases. According to another comment, the EPA should use only one type of leaching fluid for the TCLP since municipal refuse constitutes 95% of the waste present in the landfill and has the greatest impact on leachate composition. The comment goes on to say that EPA's study has shown that leachate which is generated by decomposing municipal waste contains approximately 0.14 equivalents of acidity per kilogram of dry refuse. When disposed of as a non-hazardous waste at a municipal landfill, the industrial waste tested under the TCLP will have, at the most, a minimal effect on the quality of the landfill leachate. Since the acidity of municipal landfill leachates is influenced primarily by the municipal refuse (not by the industrial wastes), the acidity of the leaching fluid should be 0.7 milliequivalent per gram of waste, as established by the Oak Ridge studies.

The Agency points out that in order for all wastes to be exposed to a leaching medium of the same pH, more acid must be added to wastes of high alkalinity. The quantity of acid used (2 milliequivalents of acid per gram of waste) is based on data collected by EPA at a municipal landfill over a period of years (US EPA, 1980). The Agency believes that, over time, the buffering capacity of alkaline wastes will be exhausted, and in order to accurately predict the leaching potential of such wastes a more acidic leaching medium must be employed. The EP test requires titration of the waste until a constant pH is reached or the maximum amount of acid is used. The TCLP requires the same maximum amount of acid for highly alkaline wastes.

One comment stated that the specified pH of 4.93 +/- 0.05 is overly precise. The +/- 0.2 pH units of the EP should prove adequate especially for organic compounds. If the pH is maintained at +/- 0.05 pH units, an option to adjust the final pH (by adding more acid or base) should be included in the procedure. Another comment stated that the pH requirement of the extraction fluid at +/- 0.05 pH units is stringent and unjustified.

The Agency asserts that, in the EP, the pH is measured in a dynamic system. Therefore, the pH measurement is maintained in a less precise fashion. In the TCLP, the pH of the extraction fluid is measured prior to contact with the waste. Adjusting the final pH should be unnecessary if the fluid is properly prepared and may cause fluctuations in the buffering capacity of the extraction fluid. The Agency believes that the requirement (in Section 5.6.1) of maintaining the pH of the extraction fluid at +/- 0.05 pH units is necessary to ensure reproducible results.

A comment was received concerning the pH of extraction fluid # 2. This comment stated that the extraction fluid should have a pH of 2.88 +/- 0.05 and questioned what should be done if it does not meet this pH requirement.

The Agency urges that the method be reviewed and that the extraction fluid be prepared again. If the fluid is prepared correctly, pH adjustment should not be necessary.

Another comment addressed the impact of the acetate buffer on subsequent biological testing. Using the acetate buffer will severely hinder the use of an integrating technique such as biological tests. This shortcoming will become more apparent as additional compounds are added to the regulated list. Therefore, EPA should study how to make the TCLP fluids compatible with these tests.

The Agency notes that no biological toxicity tests are currently used in the RCRA regulatory program. Although producing a leachate compatible with biological testing was an original objective in developing the test, the lack of such compatibility is not a serious drawback to the current regulatory program.

Several comments addressed the application of the TCLP to a variety of settings. One comment suggested that the EPA allow more appropriate extractants to be used in specific applications. This would avoid applying the TCLP to something other than its originally intended purpose. Another comment suggested that the TCLP be modified by allowing different leaching media for different uses of the TCLP. (i.e., use of distilled water extraction for purely industrial waste land disposal units).

The Agency responds that waste and disposal specific alternatives are currently being investigated.

## F. Particle Size Reduction

Fifty-one comments addressed the particle size reduction requirement. Two comments provided qualified support for this requirement while the rest were critical. Areas of criticism include particle size reduction requirements for monolithic wastes and the effect of the particle size reduction requirements on volatiles.

Numerous comments supported the re-instatement of the structural integrity procedure or some reasonable stability criterion. One comment pointed out that particle size reduction will be inappropriate in those instances where solidification of the waste is used as a treatment technique. The assumption that heavy machinery will break up the waste in a municipal landfill has never been shown to be realistic for hazardous waste management facilities. The environment inside the landfill cell (after the daily cover has been applied) would be relatively constant. This comment also stated that the structural integrity test should be retained to evaluate stabilized waste. Another comment questioned whether or not grinding represents adequately the weathering process or the vehicular traffic. It recommended that the Agency retain the SIP.

Two comments suggested improving the structural integrity test. One of these disagreed with EPA's requirement for particle size reduction and disputes the assumption that natural weathering forces are an important mechanism for breaking down monolithic wastes. This comment stated that particle size reduction could lead to a loss of volatiles or unrealistically increase the leaching rates of less volatile components. It recommended that EPA look into improving the SIP. Another comment suggested that the SIP could be improved by incorporating a compression test, a freeze/thaw test, and possibly some allowances for surface area.

Many comments agreed that particle size reduction is inappropriate for stabilized monolithic wastes and produces unrepresentative results. For example, one comment stated that particle size reduction alters the physical character of many wastes in such a way that the leaching rate increases unrealistically. This comment stated that particle size reduction destroys the cementitious property of these wastes. By increasing greatly the surface area which is available to attack by a leaching medium, the amount and rate at which substances may be leached are increased unrealistically. In as much as waste grinding is not normally employed in municipal landfills, particle size reduction renders the TCLP a less accurate model for leaching in a municipal landfill environment.

The Agency refers the commenters to the proposal in the May 24, 1988, Federal Register (53 FR 18792-18797) to use a stainless steel cage in the bottle extractor instead of requiring particle size reduction for the analysis of wastes for non-volatile contaminants. This proposal and comments received in response are discussed in more detail in Section VI of this Background Document.

Several comments suggested a standardized particle size requirement for all waste forms. Specifically, one comment recommended that the Agency incorporate particle size and surface area measurements in the protocol. Then extract concentration values should be correlated with surface area whenever comparison between waste forms are made. Another comment recommended that the size reduction requirement be revised to require a particle size which reflects a 20 to 30 psi unconfined compressive strength. According to one comment, a standardized size reduction methodology should be developed that would be used for all materials requiring size reduction.

The Agency notes that waste forms vary so widely that it would be impractical to specify a standardized size reduction procedure.

One comment stated that a minimum particle size should be specified in order to ensure uniform test results.

The Agency responds that natural or artificial particle size reduction will result in various particle sizes, and it is impractical to specify a minimum size.

Several comments which criticized particle size reduction were concerned with the effect of the requirements on volatiles. They suggested that a loss of volatiles will occur as a result of particle size reduction. One comment asserted that all reduction equipment will generate some heat, while another comment asserts that the grinding and milling equipment required for particle size reduction will result in a loss of volatiles. Another comment suggested that the loss can be substantially reduced if the sample is frozen in liquid nitrogen prior to particle size reduction.

The Agency states that it is currently considering alternatives for minimizing the loss of volatiles. However, the particle size reduction requirement will remain for volatile contaminants until additional data are obtained and evaluated. Particle size reduction is necessary to ensure contact between the leaching fluid and the waste.

Another comment stated that the Agency should allow demonstrated physical stabilization (i.e., irreversible binding to soil matrix) to be a legitimate mechanism for the environmental unavailability of contaminants.

The Agency states that if irreversible binding to soil particles occurs, the contaminants will not be found in the TCLP leachate.

## G. Procedure when Non-Volatiles are Involved

Eighty-two comments addressed issues related to the TCLP procedure for non-volatiles. Areas of concern included the sample size requirements, the filtration steps (liquid/solid separation), determination of percent solids, particle size evaluation and reduction, the alkalinity test, the extraction fluid, the filtration of the extract, the TCLP extract preparation, and the TCLP extract analysis.

## Sample Size Requirements (7.1 - 7.5)

Several comments stated that different amounts of leachate are required, depending on the analytical technique and the number of different analyses to be performed. Consequently, the actual sample size needed (not necessarily 100 g) will vary, according to the analyses which are performed. A trade organization noted that its experience with SW-846 methods for semi-volatiles, metals, pesticides, and herbicides has shown that a total of 4 liters are necessary for a complete suite of analyses. Another trade association suggested that it is improbable that a single extraction will yield enough leachate to determine all constituents. Many of the analytical methods for organics and pesticides require a liter of aqueous solution for each analysis. This will require multiple extractions and the combination of multi-liter volumes of leachate prior to analysis. Costs will increase in direct proportion to the number of extractions required to generate sufficient leachate.

The Agency recommends that, if one extraction will not generate sufficient leachate to perform these analyses, multiple extractions should be performed with the leachate from all of them combined and aliquoted for analysis. The cost will vary depending on the type of waste and the analyses required. Few wastes will require analysis for the entire list of contaminants. Process knowledge and preliminary constituent analysis (pre-screen test) can be used to determine which contaminants are likely to be present in a given waste.

Several comments expressed confusion regarding the sample size required for the procedure. They suggested different requirements for the sample size, based on the percent of solids in the sample. These comments suggested that the required solid phase sample size (75 g) is unreasonable for wastes with low percentage solids. Some comments pointed out that a waste with 1% solids would require a 7.5 kg sample and a waste with 0.5% solids would require a 15 kg sample to obtain the required sample size. A manufacturer suggested reducing the required solid phase sample size to 25g in order to reduce the volume of the original sample taken for analysis. A research lab pointed out that the required sample size of 100g yields 2 liters of leachate. However, the volume of the specified filtration vessel reservoir is only 1.5 liters. They recommended either decreasing the required sample size to 75g or modifying the filtration apparatus to accommodate the 2 liter volume. A manufacturer noted that the 75g requirement is unnecessary if a small volume of extract would support the analyses. It recommended that the wording be changed to state that enough solids must be generated for extraction to support analyses.

The Agency agrees with the last commenter and has changed the method requirements to indicate that enough solids must be generated for extraction such that the volume of extract will be sufficient to perform the required analyses. The method recommends a minimum of 75g of solids if the full range of analyses is required. In most cases, the extract will not need to be analyzed for the entire list of contaminants.

## Filtration Steps - Liquid/Solid Separation (7.7)

Some of the comments in this section addressed the failure of the TCLP to provide directions as to how fluids from centrifugation are to be treated. One comment questioned whether these fluids should be discarded or combined with the fluid expressed from the wastes in step 7.9. If the latter is correct, they suggested that section 7.16 be expanded to include an equation that includes the amount of waste originally centrifuged, volume of fluid obtained by centrifugation, amount of waste charged to the extractor as a percentage of the waste originally centrifuged, and the amount of centrifuged liquid analyzed (separately or combined) as a percentage of the total fluid obtained. An oil company believed that centrifugation should be allowed as a laboratory technique for separating and evaluating wastes when volatile compounds are not of concern. Centrifugation would eliminate problems such as filter plugging and help to achieve acceptable separations.

The Agency agrees that clarification of the method is required regarding the use of centrifugation. The method now states that centrifugation is to be used only as an aid to filtration and only when non-volatiles are the analytes of concern. Following centrifugation, the liquid should be decanted and filtered, followed by filtration of the solid through the same filter system. The instructions have been moved to the front of the method for non-volatiles. (See Step 8.6)

Another trade association suggested that, to remain consistent with the original design of the EP where particles greater than 0.45 um were excluded, the TCLP should allow for the clarification or removal of fine particulates which might pass through the less selective glass fiber filters. It suggested using 0.45 um membrane filters as in the EP. Including these fine particles adds the extra burdensome step (as described in step 7.16) of acid digestion when analyzing for metals. This acid digestion of particulates would yield an obvious increase in metals concentration if metals were indeed present in the solid waste and is not representative of the leached concentration of metals.

The Agency believes that particles which pass through the filter may also migrate with the leachate. Release of metals from ligands is critical and the acid digestion of the leachate prior to analysis is necessary in order to accomplish this.

A few comments addressed the actual filtration step itself and how the decision to stop applying pressure should be made. One manufacturer recommends that the filtration step which specifies that the filtration pressure should be increased until "no additional liquid has passed through the filter in any 2 minute interval" be clarified. Also, replacing the phrase "no additional liquid" with a relative volume such as 1% would eliminate further confusion. A contract laboratory requested further clarification of the directions which state that "no fluid is obtained under pressurization."

EPA believes it will make little difference whether "no additional liquid" or "1% in a two minute interval" is specified (to define the termination of filtration). However, it should be easier to detect the cessation of flow rather than to quantify continuing flow. Therefore, the instruction in the method has not been changed.

Other comments refered to transferring the sample from the shipping container to the filtration device and suggest a way of diminishing sample loss. A trade association claimed that problems with the adherence of waste to the walls of the container can be avoided if the waste is transferred directly from the waste shipping container into the filter holder. The amount of waste charged is equal to the weight of the original container minus its weight after the waste is transferred. Two manufacturers stated that clarification is needed regarding when a correction to the original sample weight should be made. Such a correction could change the solid to liquid ratio and consequently have an impact on the characterization of the waste. They recommended that the existing correction note be replaced with an emphasis on the quantitative transference of sample to filtration device.

The Agency has changed the method directions to emphasize a quantitative transfer of solids. The note states that, if any solid (> 1%) has adhered to the filter holder, the person conducting the test should determine the weight of this solid and subtract this weight from the amount of solid in the filtration device before transfer. (See Note at Step 8.7)

Another trade association comment stated that acid and distilled water filter rinsing (as required in step 4.4 for filters used in preparation for metal analyses) should be expanded and allowed when evaluating the mobility of all non-volatiles.

The Agency states that its ruggedness study does not indicate a need for cleaning filters for non-metallic samples. However, the method does not prohibit acid and water rinsed filters for all non-volatile analyses.

A municipality stated that more than one filter should be allowed in step 7.7 to lessen the likelihood of rupture and degradation of the glass fiber filter (possibly caused by instantaneous application of high pressure).

The Agency has added a second note in Step 8.7 (formerly 7.7) indicating that pressure should be increased gradually to minimize the likelihood of rupturing the filter.

One commenter was concerned that the instructions in step 7.7 imply that vacuum and pressure filtration are equivalent. The instructions should specify that pressure filtration is necessary unless air moves through the filter at 10 psi.

The Agency agrees. Vacuum filtration is recommended only for waste with low solids content (<10%) or for highly granular waste. See Step 4.3.2.

## Determination of Percent Solids (7.9 - 7.10)

One comment suggested an increase in the percent solids required to discard the solid portion from 0.5% to higher levels, e.g. 1%. Several comments recommended removing this portion of the procedure altogether for reasons discussed below. A state agency noted that the EPA criterion for performing an extraction is the weight of residue which is collected on a filter disc when 100 ml of sample is filtered. If the weight of the residue is less than 0.5 g, the filtrate is used as the extract for elemental or compound analysis. The comment expressed disagreement with the cutoff amount of 0.5 g and believes it should be increased to at least 10 g. The comment suggested that a better evaluation of mostly liquid samples would consist of pouring off the liquid, weighing 100 g of solid sample, re-combining the liquid portion of the sample, adding the extraction fluid, and performing the TCLP extraction procedure.

A municipality suggested that, instead of filtering the original waste, one could calculate the amount of leaching solution to add to the original waste. The amount added by weight would be 20 times the weight of the bone dry solids in the sample. In this simplified, one time filtration procedure, the ratio of leaching fluid volume to total solids would be less than or equal to that ratio in the procedures as written. This method would provide for a worst case situation and expedite matters especially where volatiles are concerned.

Another municipality pointed out that, in the test procedure (step 7.10.4), wastes with less than 0.5% solids are filtered. The liquid phase is then used as the TCLP extract. For wastes with more than 0.5% solids, the solid phase of the waste is diluted 20:1. The comment suggested that since liquid wastes are also diluted and attenuated in the environment, liquid wastes (less than 0.5% solids) should also be subjected to the 20:1 dilution factor. A federal lab also suggested that the pre-extraction separation procedure be dropped and the entire original sample extracted at a 20:1 ratio. This would help eliminate the loss of volatiles and facilitate only one extraction for all concerned constituents.

A chemical company stated that the initial solid/liquid separation adds an unnecessary step to the entire TCLP. This step creates some manipulative problems with the arbitrary classification of wastes based on 0.5% solids content. It suggests that the test consist of simply mixing 25 g of sample into 500 ml of extract solvent, contacting for the desired time, filtering off enough liquid for analysis, and proceeding with the GC/MS test. Including the less than 0.5% solids for extraction is of no consequence while eliminating steps 7.9 through 7.10 will save significant amount of time and money.

The Agency responds that the solid/liquid separation has been shown important in the extraction of metals and semi-volatiles. The Agency is investigating the elimination of the solid/liquid separation step for volatiles.

Several comments suggested that a precautionary note be added which states that the drying oven should be vented to a hood or other appropriate device. They noted that an explosion may occur when certain types of organic materials (i.e., polynitro aromatics) are taken to dryness.

The Agency has had no reports of explosions, but agrees that a cautionary note is in order and has added one to the procedure. (See Note at Step 7.2.2.)

A commercial laboratory pointed out that in the TCLP procedure there are four different methods for determining percent solids. By using the TCLP procedure, a different percent solids would probably be obtained on the same sample when extracted for both volatile and non-volatile components. It recommends that a single method for determining percent solids be required. The piston-pressure filtration would be a good choice for this. However, that method would require that all labs possess a ZHE, even if they only analyze for non-volatile components. Therefore, in order to eliminate unnecessary costs (i.e., purchase of ZHE), they recommended use of the vacuum filtration method.

The Agency has re-written the method instructions regarding determination of percent solids. This procedure only must be performed once, and the results may be used for the determination of both volatile and non-volatile analytes. (See Section 7.1) Non-ZHE pressure filtration devices are available, and such devices are recommended for wastes with greater than 10% solids unless they are highly granular. (See Section 4.3.2)

A state agency noted that no provision is made for samples containing exactly 0.5% solids. They recommended that the wording be changed to "for waste containing 0.5% solids or greater."

EPA notes that step 2.2 has been changed to indicate greater than or equal to 0.5% solids.

A trade association stated that solids should be determined on a "dry" basis. As written, the test would allow varying degrees of moisture in the solid portion of the waste. This would result in varying degrees of active ingredients in the waste samples that are subject to the actual extraction. This will create inconsistent analytical results among different laboratories.

The Agency replies that the method is based on an extraction of a sample of the waste that is not filterable under the conditions of the test. It is not meant to extract "dry" solids only, since wastes are not landfilled in a "dry" condition. The portion of the waste which will not pass a 0.6 - 0.8 um filter under 50 psi is assumed to correspond to the waste in the landfill which will be subject to leaching.

## Particle Size Evaluation/Reduction (7.11)

Two comments received regarding particle size evaluation/reduction asserted that particle size reduction should be accomplished prior to step 7.5 (to avoid sample loss during the grinding step). They noted that moist material will adhere to the particle size reduction equipment. Such a loss will be impossible to measure.

EPA agrees with these comments and has made this change. It is believed that this will result in a smaller loss of analytes and in a maximization of sample homogeneity in the procedure. (See Step 6.2)

Another comment stated that procedures for particle size reduction and surface area measurement must be specified in order to assure equivalent results among analysts and among laboratories.

The Agency responds that any particle size reduction technique will result in various sizes of particles. Therefore, a common technique will not necessarily provide reproducible results. Also, materials which require particle size reduction (i.e. cement blocks, telephone poles, steel beams, etc.) vary so much that it would not be practical to specify a single technique.

## Alkalinity Test (7.12)

Two comments suggested that the size of the subsample used for the alkalinity test (step 7.12.1) (on a dry weight basis) be specified. The amount of acid added in step 7.12.3 is 3.5 millimoles. This is equal to 140 mg of NaOH. Therefore, if the subsample is small enough, all wastes will be determined to require extraction fluid #1. The comments suggested that the only way to avoid this problem is to decide on a total alkalinity cutoff value for the waste solution generated in step 7.12.1. This will increase the amount of time and money required to perform the TCLP.

The Agency replies that the method specifies a 5.0 gram sample of the solid phase of the waste be used for the alkalinity determination. The solid phase is that portion which will not pass a 0.6 - 0.8 um glass fiber filter under a pressure of 50 psi. The quantity of acid required is 3.5 mL of 1N HCl, or 3.5 millimoles. (See Step 7.4)

A manufacturer believed that requiring particle size reduction to 1 mm for the alkalinity test is contradictory since step 7.11 requires a reduction to only 9.5 mm. The comment also questions the justification for including Steps 5.6.1 and 7.12.1 through 7.12.4. The comment asked for clarification regarding how the specific volumes, weights, time, temperatures, and normalities were determined and recommended that Step 2.0 (the method summary) include some explanation of the above mentioned steps.

The Agency responds that the 1 mm particle size is designed to demonstrate quickly and easily the buffering capacity of the waste and to ensure that the 5 g aliquot is as representative as possible. Additional information is provided in ERCO, 1986, and USEPA, 1986a.

### Extraction Time (7.13)

Several comments discussed the requirement of an 18-hour extraction time. A chemicals and metals processing firm supported the reduction in extraction time from 24 hours (for the EP) to 18 hours (for the TCLP). Several trade associations believed an 18-hour extraction time creates problems in scheduling laboratory work. They suggested that some tolerance interval be allowed. One comment stated that this time period restricts a typical laboratory to beginning the test in the afternoon and completing it the next morning. This would result in potentially unnecessary logistical problems and labor costs. Maintaining the 24-hour (plus or minus four hours) extraction period would coincide more readily with a typical work schedule. Another asserts that unless EPA has data showing that the exact length of agitation period is critical, the required agitation period should include a tolerance interval (i.e., 18 +/- 2 hours). This comment also requests an explanation of the basis for choosing 18 hours as an agitation period.

The Agency agrees with the comments regarding extraction time. It has changed the method to read 18 +/- 2 hours, based on the ruggedness testing. The Agency does not believe that the 18 hour extraction time disrupts work schedules more than the 24 hour extraction period does. Discussions of the effects of changes in the time of agitation can be found in US EPA, 1986a, and Brown et al., 1983.

## Filtration of Extract (7.14)

Several comments stated that the directions for the re-combination of the filtered extract and the filtrate from the initial liquid/solid separation do not consider the reduction in the amount of solid extracted due to performance of the alkalinity test and any adherence of solids to the wall of the filtration vessel. The comments pointed out that the volume of initial filtrate should be reduced proportional to the loss of solids.

The Agency replies that the alkalinity test is now performed on a separate sample of the waste, and emphasis is placed on the quantitative transfer of solids from the filtration device to the extraction vessel. Adjusting the volume of the initial filtrate to account for loss of solids would further complicate the method with a negligible impact on results.

A research laboratory suggested that, following extraction of the solid waste and filtration of the leachate, a preservation step should be added (instead of referencing a procedure in SW-846). It recommended that a table be made which clearly states the preferred method of preservation and holding times for each measured parameter.

EPA has changed the method to specify that aliquots of leachate for metals analysis be acidified to pH 2 with Nitric Acid as soon as possible. Other non-volatile and volatile samples should be refrigerated at 4°C until analyzed. See Step 8.14. Holding times prior to analysis are addressed in Step 10.6.

A trade association questioned whether or not it is necessary to filter the extract if a non-filtered extract passes the test.

The Agency agrees that a non-filtered extract will probably contain higher concentrations of analytes than a filtered extract. Analyzing unfiltered extract is not part of the method. A pre-screen test, consisting of a total waste analysis, has been included as part of the method for persons who want to demonstrate that the waste itself does not contain hazardous levels of the regulated constituents.

One comment stated that filtration presents a real problem for tars, viscous polymer wastes, and still bottoms that are fluid only at high temperatures.

The Agency agrees that these materials do offer special analytical difficulties, whatever approach is taken. It is likely that the TCLP would treat these samples as 100% solids. The behavior of difficult to filter wastes in the TCLP is currently being investigated.

TCLP Extract Preparation (7.16)

Some comments stated that the volume of initial fluid expressed from the waste may be insufficient for analysis by SW-846 methods. If this is the case, the comments questioned whether the fluid may be diluted to a volume sufficient to meet the analytical requirements or how the fluid must be analyzed.

The Agency responds that if insufficient leachate is obtained, the procedure may be repeated until enough liquid is available to perform the analyses. In some cases, large quantities of sample and multiple extractions may be required. Dilution of the leachate is not permitted, because dilution may reduce the concentration of contaminant in the fluid being analyzed to a level below the limit of quantitation of the analytical procedure.

A state agency noted that Part 3.0 refers to analytical interferences. However, there is no mention made of potential interferences with the leaching procedure itself. The writer suggested that possible acid-base reactions, gas evolution, and problems with oily waste should be discussed in Section 7.0.

The Agency does not consider acid base reactions, gas evolution, or oily waste to be interferences in the procedure. Notes on how to handle such occurrences can be found in sections 8.8, 8.11, and 9.10.

A trade association questioned whether or not the acidity (pH) of a leachate should have a limiting value. It suggested that a waste which generates a low pH leachate would seem undesirable in itself.

EPA's corrosivity characteristic addresses wastes with extreme pH values.

## TCLP Extract Analysis (7.16)

A number of comments discussed analytical procedure issues. A trade association noted that Method 8280 for dioxin analysis was mentioned in the January 14, 1986, proposal as being part of SW-846. However, it has never been included in the SW-846 manual. A metals processing firm recommended that other analytical procedures which are equally valid be approved as alternatives. It also recommended that the decision of which method to use be left to trained analytical chemists. A chemical company believed that the high variability of analytical methods is a matter of concern. Since the precision of the TCLP is affected by both the extraction procedure and the analytical methods, the variability of the TCLP would raise some questions in the use of the procedure for enforcement purposes.

The Agency notes that, where alternative methods are available within SW-846, the analyst has the option of selecting among these for compliance with the requirements of the Toxicity Characteristic. Method 8280 is included in the Third Edition of SW-846. In addition, data on alternative methods can be supplied to EPA for possible inclusion in future editions of SW-846. EPA believes that the variability of the method can be reduced by multiple extractions and compositing of extracts prior to aliquoting for analysis. Multiple sub-sampling will reduce the variability in the TCLP.

Several comments voiced concern about the analysis of TCLP extracts for metals. A trade association stated that, if leaching metals are of prime concern, acid digestion is not needed for most atomic absorption spectrophotometric analyses. If release of metals from ligands for analysis is deemed critical, then the comment recommended that the acid and the conditions of digestion be specified. A state agency recommended that the directions be changed to read "TCLP extracts to be analyzed for metals other than mercury be acid digested" because no acid digestion is involved in the analysis of mercury.

EPA replies that acid digestion is needed for the TCLP prior to analysis for metals other than mercury. The metal content of particles and release of metals from ligands is critical. The third edition of SW-846 gives explicit instructions on necessary procedures. Mercury, however, need not be acid digested, and the method has been changed accordingly. (See Step 8.14)

One waste management firm claimed that if extremely small particles pass through the filter and the filtrate (with the small particles) is subjected to a flame method of analysis, the particle concentrations (as well as the leachate concentrations) will be detected. The TCLP is supposed to analyze the leachate alone, not falsely determine solid concentrations due to the inadequacies of the procedure.

The Agency responds that the method is intended to measure metals which are mobile in the environment. Particles which pass through the filter are assumed to migrate with the liquid in the subsurface and pose a potential threat to ground water.

Another trade association believed that a compatibility test must be described. The formation of a precipitate should not prohibit combining liquid phases in order to determine inorganics. The subsequent SW-846 analytical procedures require digestion prior to analysis, thus making the formation of a precipitate irrelevant.

EPA replies that "compatibility" for the purpose of this test includes immiscibility as well as formation of precipitate. No test for compatibility will be described, because a visual determination will be sufficient in most cases. The method has been changed to state that, for inorganic analysis, if a precipitate forms upon combination of the liquid phases, this does not constitute incompatibility since the extract must undergo acid digestion prior to analysis. (See Step 8.13.2)

A trade association recommended that, although it is not necessary to measure pH and alkalinity of the TCLP extracts, such measurements should be taken as the Science Advisory Board recommended in 1984. These two measurements are often useful in deciding whether or not measured concentrations in the extract are chemically consistent with the solution characteristics. These measurements are easy to take and can provide quality assurance checks for the measured concentrations.

The Agency agrees with the commenter that such data are necessary for proper interpretation of the results and has revised the method accordingly. See Step 8.14.

One comment requested information on the non-volatiles that should be analyzed, on the procedures that should be followed, and on the allowable concentration limits that apply. In addition, a trade association claimed that Step 7.16 should specify the methods of analysis to be conducted on the extracts.

EPA points out that the TCLP, Method 1311, is used for several regulatory purposes. Therefore, it is not appropriate to include instructions as to what analytes should be measured in the method itself. The analytes of interest the Toxicity Characteristic and the analytical methods required for compliance with this regulation can be found in 40 CFR 261.24.

## H. Procedure when Volatiles are Involved

The Agency received fifty-two comments which addressed issues such as sample size requirements and determination, particle size evaluation and reduction, filtration in the ZHE, determination of the solid phase, the extraction fluid, the TCLP extract preparation, and the TCLP extract analysis. These comments are summarized and addressed below.

### Sample Size Requirements and Determinations (8.1 - 8.4)

A commercial laboratory pointed out that one would need a 5 kg sample to obtain 25 g of solid if the solid content is 0.5% of the total waste.

The Agency has revised the procedure to indicate that enough solids must be generated for extraction such that the volume of extract obtained will be sufficient to perform the required analyses. The 25 gram sample size is a maximum, not a minimum.

A commercial laboratory also suggested that analytical laboratories performing TCLP analyses request duplicate samples, using one to determine both which extraction fluid to use and the percent solids and using the other for the actual extraction. The comment asserted that this would minimize the loss of volatiles from the sample before the extraction is begun.

EPA agrees with the comment and has changed the method to this effect. See Section 7.0 - 7.5.

One manufacturer noted that, for a complex waste, it will be necessary to use the procedures for both non-volatiles and volatiles to properly characterize the waste. In such cases, it recommended that one should have to determine the percent solids only once. (See Steps 7.0-7.6.4 )

EPA agrees and has changed the method to stipulate that percent solids is measured using the hazardous waste filtration apparatus and the measurement applied to both the ZHE and the conventional apparatus.

A trade association felt that the percent solid should be determined on a "dry" basis because variation in the moisture content would also result in variation in the contaminant concentration.

The Agency replies that the method is based on an extraction of a sample of the waste that cannot be filtered under the conditions of the test. It is not meant to extract "dry" solids only, because the wastes as disposed in a landfill contain moisture.

Another comment stated that volatiles are always lost from samples. Thus, the statement that reads "care must be taken to ensure these (i.e., volatiles) are not lost," should be changed to reflect the fact that losses can only be minimized.

EPA agrees and has modified the method, as suggested. Refrigeration of samples and equipment is suggested in order to minimize loss of volatiles.

Particle Size Evaluation/Reduction (8.5)

In reference to particle size evaluation and reduction, some trade associations believed sample size reduction should be performed before the analytical sample of waste is weighed out. This same point was made in 7.11. Loss of sample is unavoidable during particle size reduction. Therefore, it should be done on the largest size sample of bulk waste. The comments suggested that this will minimize the losses on the final analytical sample.

EPA agrees and has made this change. It is believed that this will result in minimizing loss of sample and in maximizing sample homogeneity. (See Step 9.0.)

Another trade association suggested that particle size reduction should be done in the laboratory after the refrigerated transport and storage of the sample to prevent loss of volatiles. This should be done since the laboratory is usually a cooler and more controlled environment than the field. A commercial laboratory suggested that liquid nitrogen freezing, followed by fracturing, be considered as a size-reduction technique. It claimed that the liquid nitrogen would minimize the loss of volatiles, make most samples brittle and easy to fracture, and would not contaminate the sample.

The Agency notes that the method encourages cooling of the sample prior to particle size reduction to reduce loss of volatiles. The method recommends particle size reduction in the field, but the decision on how to handle specific wastes is left to the analyst.

Another comment suggested that sieving samples to evaluate particle size would lead to loss of volatiles since the sample would come into extensive contact with air.

EPA agrees that particle size reduction for volatiles is a difficult issue, and several alternative approaches are being investigated. In the meantime, EPA is recommending that a sieve not be used to determine particle size for volatiles. A small ruler is an acceptable alternative and its use is suggested until a more adequate method is developed.

## Filtration in ZHE (8.7-8.8)

One manufacturer recommended that the note associated with the correction to the original sample weight to account for loss of sample during transfer to the ZHE be replaced with an emphasis on the quantitative transference of the sample to the filtration device. Such a weight correction could alter the solid to liquid ratio of the sample and have a significant impact on characterization of the waste.

EPA has revised the method as suggested to emphasize quantitative transfer of the sample. See Section 9.8.

A trade association asked whether it is necessary to use a pump to transfer extraction fluid into the ZHE.

The Agency responds that it is not necessary to use a pump. One alternative to a pump is to use another ZHE or pressure filtration device to transfer extraction fluid into the extraction vessel. The procedure is outlined in Section 9.12.

Two comments pointed out that any fixed volume collection container used in step 8.8 will contain headspace after the fluid is expressed from the waste. If the volume of the container matches the volume of the expressed fluid, this would be entirely fortuitous. Small volumes of expressed fluid can be collected in gas-tight and PTFE syringes with fluid locks on the inlets and stored in this manner. Tedlar bags are the only acceptable containers for large volumes, and so the method should require them.

The EPA agrees and has changed the method to recommend the use of Tedlar bags when the waste contains an aqueous liquid phase which subsequently must be combined with the leachate. If the waste contains a non-aqueous liquid phase, either a syringe or a Tedlar bag may be used. Refer to Step 4.6 for more details.

A trade association suggested that the evacuated container which will receive the filtrate be more fully described and the sample weighing method which minimizes the loss of volatiles be specified.

The Agency points out that Tedlar bags or gas-tight syringes are specified in Step 4.6 as appropriate containers to receive the filtrate from the ZHE. It is not necessary to specify a degree of evacuation or internal pressure for these devices. The analyst is instructed to take the necessary precautions to minimize the headspace in the filtrate collection containers. No single weighing technique would be appropriate for the variety of wastes analyzed by this method, so the selection of appropriate weighing procedures is left to the analyst.

A commercial laboratory recommended that all filtration be done with the ZHE in an upright position with the filter on the top. At the completion of the extraction, the vessel should be placed in this position and the solids allowed to settle for 5-10 minutes prior to the final filtration. This procedure will minimize clogging of the filter during the filtration. As a quality control step, the commenter also suggests that the Tedlar bag or gas-tight syringe be weighed after the final filtration. This would indicate whether or not all of the extraction fluid is recovered.

The Agency agrees. Specifying that filtration be performed with the filter on top will ensure that results are more reproducible. Step 9.8 specifies that filtration in the ZHE must be carried out with the gas inlet/outlet flange on the bottom and the liquid inlet/outlet flange (with the filter) on top.

Another comment asked why the limit of 50 pounds per square inch gauge applied pressure was specified for filtering samples.

EPA refers commenter to the March 10, 1986, TCLP background document for a discussion of the rationale behind the filtration pressure of 50 psi (US EPA, 1986a). The pressure has been reduced from the 75 psi used in Method 1310 (EP) because the glass fiber filter has a larger pore size than the membrane filter and thus has less of a tendency to clog, allowing a reduction in the maximum pressure.

A manufacturer recommended that the filtration step (which states pressure is to be increased until no additional liquid has passed through the filter in any 2 minute interval) be changed to a relative volume change of 1% to be measured by weight. This suggestion was made because of the difficulty in detecting flow into the Tedlar bag.

The Agency believes that it is easier to detect a cessation of flow than to quantify a relative volume change. Therefore, the method has not been changed in this regard.

Another comment noted that Section 4.3.1 refers to the use of in-line filters when the filter within the ZHE breaks. The comment asked for a definition of these filters and suggests that the filter be specified as other filters are specified and manufacturers indicated.

EPA responds that this equipment is commonly available from instrument supply catalogs.

## Determination ~~of~~ Solid Phase (8.9 - 8.10)

A trade association asserted that the note given in Step 8.9 addressing difficult to filter wastes should be moved to Step 8.4. Another trade association suggested that the term "100% solids for the procedure" as written be defined as: that fraction of a sample from which no liquid may be forced out by an applied pressure of 50 psig.

The Agency agrees with both comments. The information in the referenced note is specified in Steps 7.1.8, 8.8, and 9.10. The solid phase of the waste is defined as the material in the ZHE following the initial filtration, also in the three sections referred to above.

Extraction Fluid (8.11)

One comment stated that using only extraction fluid No. 1 in the ZHE avoids the problems of bias due to the difference in solubility of acidic and basic compounds at different pH values. However, when extraction fluid No. 2 is used in the extraction for metals and semi-volatile organics but is not used in the ZHE portion of the test (for highly alkaline wastes), the volatiles analysis will show a higher level of acids (relative to base/neutrals) than that of the semi-volatiles. It is conceivable that a waste generator could "treat" his waste by adjusting the pH level to the point where the concentration of contaminants in the extract will be below the regulatory threshold simply by forcing an external bias on the test instead of improving the overall nature of the waste.

EPA notes that the extraction of volatiles is not greatly affected by changes in the pH of the extraction fluid. Adjusting the pH level of the waste would be considered waste treatment and requires a permit.

A trade association noted that the TCLP procedure requires extraction fluid number 1 be used with the ZHE. Fluid number 2, however, is specified for metals in high alkaline waste. The comment asked if ZHE and metal analysis are required on an alkaline waste, will two separate extractions be required? The comment maintained that this would not be cost effective.

The Agency reiterates that the ZHE procedure is used only for determining volatiles in the leachate. Semi-volatiles and metals analysis must be performed on leachate obtained with the bottle extraction. EPA believes that this is cost effective, as discussed in Section I of this document.

A manufacturer recommended clarifying Step 8.11 to indicate that the temperature external to the extractor is to be controlled. Another comment asked whether the temperature of 22 +/- 3°C during agitation refers to ambient air temperature or temperature of the contents of the ZHE.

The Agency responds that the specified temperature refers to ambient air. The method has been clarified on this point. See Step 9.12.3.

One trade association noted that, if the regulatory thresholds for the Toxicity Characteristic are below the saturation concentrations for each component, then the use of a volume of extraction fluid less than 20 times the weight of the 100% solids sample would seem allowable. If the waste passed this worst case condition, then the waste would pass the more dilute test.

EPA observes that, while this may be true, co-solvent effects may produce concentrations greater than the water solubility. Standard procedures are necessary to permit comparison of results between laboratories.

## TCLP Extract Preparation (8.12 -8.14)

Several comments discussed multiphasic leachates. One trade association noted that the method requires prior knowledge of the waste to predict if multiphasic samples will be generated from the wastes when the initial liquid phase is combined with the leachate. Another trade association made the same comment and suggests that the TCLP describe a screening test to guide the analyst in the choice of using a separate filtrate container to hold the leachate. A manufacturer believed EPA should provide guidance for resolving problems presented by multiphasic filtrates.

The Agency believes that generators should have sufficient knowledge of their wastes to predict the occurrence of multiple phases in the filtrate and extract. Such multiphasic samples may be analyzed separately and the results mathematically combined to obtain the total concentration of contaminants. See Step 9.15.

A municipal sewage authority experienced some difficulty with using Tedlar bags for transferring sample filtrate and extract from the ZHE. The comment noted that some amount of air invariably makes its way into the bag.

The Agency notes that any collection device will contain some air. However, the Tedlar bags have been shown to minimize the amount of air present.

A state agency recommended modifying the description of the procedure for checking the ZHE to be sure that pressure had been maintained during the extraction procedure. It claimed that a liquid release would also indicate that pressure had been maintained.

The EPA has specified that the ZHE should be checked for leaks after every extraction by pressurizing to 50 psi and submerging it in water. See Step

4.2.1. Step 9.13 instructs the operator to check for release of gas from the gas inlet/outlet valve following the 18 hour agitation period. No release of liquid should occur there.

TCLP Extract Analysis (8.14)

A metals processing firm recommended that other analytical procedures which are equally valid as those included in SW-846 be approved as alternatives. Trained analytical chemists should decide which method to use.

EPA responds that existing regulations allow companies to submit procedures for consideration as an equivalent. Users are encouraged to submit data on alternative procedures to the Agency for possible inclusion in future editions of the SW-846.

## I. Quality Assurance Requirements

The quality assurance requirements for the TCLP are more complex than those for the EP and include a minimum of one blank for every ten extractions done in the extraction vessel to check for memory effects from the extraction equipment. Other QA requirements include the method of standard addition for each metallic analyte if either the recovery of the compound from the TCLP extract is not between 50 and 150% or if the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more than one extraction is run on samples of the same waste, the method of standard addition may be applied only once and the percent recoveries applied to the remainder of the extractions. A third QA requirement involves the holding times for the samples. The period that an extract may be held before extraction and between extraction and analysis is 14 days for volatiles, 40 days for semi-volatiles, 28 days for mercury, and 180 days for other metals.

Twenty-two comments expressed concern about these quality assurance requirements. Two comments requested that EPA specify which extraction fluid should be used as the method blank when the extractor is checked for memory effects.

EPA replies that Extraction Fluid #1 should always be used for blank determinations, in order to ensure consistency and comparability.

Another comment noted that the requirement of one blank for each batch of extraction fluid means that a blank must be run each day. This will require a large percentage of ZHEs from most laboratories. The comment asked if the extraction blank can be run directly as prepared or be analyzed only if the contaminant measures above the regulatory limit. Another comment claimed that the blanks and samples cannot be run the same day because of the eighteen hour extraction time requirement. A major oil company questioned whether or not the analysis of blanks on a 10% basis will be adequate if significant sample carryover is observed.

The Agency responds that good laboratory procedure requires a blank for each batch of samples. This requirement is a minimum. More blanks may be run, and should be run if a problem is suspected. Most laboratories conducting the analysis have multiple extraction vessels, and the vessel used for blank analysis should rotated on a regular basis. The blank should be carried through all steps of the procedure at the same time as the samples. The method no longer requires that the extraction fluid be made up daily.

Several comments addressed the subject of standard additions. Two comments concurred with EPA's approach to standard additions. Many comments requested EPA to specify whether all possible contaminants are to be determined by Method of Standard Additions or just the ones found above the method detection limit. A major petrochemical company expressed several concerns with the specifications for spiked standards. It questioned whether it is reasonable to require a 50-150% recovery for all spikes. Another comment stated that it may be difficult to achieve 50% recovery of phenolics spiked into samples. Also, the MSA requirement means that a minimum of two spiked samples, an unspiked sample and a blank be run for each determination. Finally, it asked that EPA specify when the spikes must be added (either before or after extraction).

The Agency points out that step 10.5 now states that the method of standard additions (MSA) must be performed for each metallic analyte if recovery is not between 80% and 120% or the measured concentration is within 20% of the regulatory threshold. MSA is not required for organic analytes. Step 10.3 specifies that the spikes are added after filtration of the TCLP extract and before preservation since the purpose is to evaluate the analytical method's capacity to measure the concentration of the contaminant in the extract. Blanks are required for each analytical batch (up to 20 samples).

One comment noted that no definition of "spiked splits" or mathematical analysis of "recovery" appears in Section 9.4. A laboratory suggested the use of surrogate spikes. Another comment suggested that, for organic constituent analysis, methods should be modified to allow isotope dilution instead of standard addition and that a single addition technique be allowed instead of multiple standard additions.

The Agency has deleted the reference to "spiked splits", and believes a mathematical analysis of recovery is not necessary. Recovery should be reported in terms of the analyte and material spiked. Surrogate spikes are not required because the purpose of the method is not to be an efficient "extraction" but to represent leaching potential in the field. Isotope dilution methods are expensive and cumbersome, and have not been approved for RCRA analyses.

Two comments suggested setting a time limit for the holding time for semivolatile samples. One also recommended holding times of 7 days for semivolatile samples and 14 or 21 days for metal analysis.

The Agency notes that holding times both before and after extraction are now specified in step 10.6.

An industry trade association contended that the quality control requirements of the TCLP will greatly affect the cost and productivity of the procedure.

The Agency emphasizes that the quality control requirements are essential to ensure the reliability and appropriate interpretation of results.

Other comments requested guidance for the time for retaining analytical and QA data. One of these comments suggested a minimum of 3 years in order to be consistent with the RCRA programs.

The Agency notes that data retention requirements are not a part of the method, and are therefore not addressed in this background document. Such requirements are, however, part of many regulations. The preamble to the final rule should be consulted on this point.

## J. Relationship of the Multiple Extraction Procedure (MEP) and Oily Waste Extraction Procedure (OWEP) to the TCLP (Method 1311)

The Multiple Extraction Procedure (MEP) has been used to predict the long-term effects which acid rain may have on stabilized wastes. The Oily Waste

Extraction Procedure (OWEP) has been used to predict the leaching of metals from wastes which contain significant amounts of oily materials. Both methods have been used to make waste specific determinations under the listing and de-listing programs.

Six comments were received which addressed this topic. Two comments supported continued use of the MEP and OWEP for the de-listing program. One of these comments also supported using the procedures for the listing of hazardous wastes and recommends that EPA allow the regulated community to submit additional alternative extraction procedures for specific wastes and disposal practices. A major oil company recommended additional testing of the mobility of oil in the disposal scenario. Another comment recommended testing inorganic stabilization treatment options which may be evaluated using the MEP. A natural gas company contended that the analytical results for metals will differ significantly between the TCLP and OWEP.

The Agency replies that it will continue to employ the MEP and OWEP only in the listing and de-listing programs where situation-specific decisions can be made. The TCLP will be used to determine if a waste is hazardous, according to the Toxicity Characteristic (40 CFR 261.24). Further studies are underway to investigate whether or not changes to the TCLP can enhance the test's ability to model the behavior of oily wastes in a land disposal environment.

## K. Other Technical Comments

This section contains miscellaneous technical comments concerning the TCLP. Some of these comments are very general while others are very specific.

The most frequent general technical comment was that the procedure is too complicated. Several made suggestions to make the procedures easier to understand and perform. Another comment suggested separating the procedure into two separate documents, one for volatiles and one for non-volatiles, to make the procedures easier to understand.

The Agency believes that the changes in the procedure resulting from EPA's reevaluation and the comments received have made the procedure easier to understand and perform. Changes are documented in a subsequent section of this report.

Sampling guidance was another topic that elicited multiple comments. Three comments requested guidance in obtaining representative samples. One comment requested that the procedures provide methods for determining sampling frequency and replication, as well as an indication of the appropriate measure for comparing with the regulatory level.

EPA replies that additional sampling guidance is provided in Chapter 9 of the third edition of SW-846. The Agency is currently developing a more detailed guidance on appropriate sampling measures.

A trade association recommended a three year sunset provision to enable EPA periodically to update the protocol as new analytical tools are developed.

The Agency disagrees. Monitoring and updating of the procedures are constantly being performed by the Agency.

## VI. Comments on May 24, 1988, Proposed Modifications

In response to comments on the particle size reduction requirement, the EPA reviewed the use of the Structural Integrity Procedure, which employs a drop hammer to test the integrity of solidified and monolithic wastes. The Agency found that certain materials maintained their integrity in the SIP, but when they subsequently were placed into the glass extractor bottles and rotated the bottles would break. The Agency developed a cage insert for the bottles in order to prevent breakage when solidified or monolithic materials were extracted. While evaluating the utility of the cage to prevent breakage of the bottles, the Agency noticed that wastes that were believed to be well-solidified retained their monolithic nature in the cage during extraction, whereas wastes that were believed to be less well-stabilized broke into small pieces.

The Agency conducted further evaluations, as discussed in the May 24, 1988, Federal Register notice, 53 FR 18792 - 18797, and preliminary results suggested that the cage tumbling procedure may correlate better with the environmental stability of the waste than the SIP. Based on these results, the Agency proposed that most waste materials need not be milled to pass the 9.5 mm sieve before testing if (1) the bottle extractor equipped with the cage is employed and (2) an appropriate size representative sample can be taken and analyzed. The exception would be wastes that are rendered monolithic by being encapsulated and wastes that are tested for volatiles.

Eight commenters indicated that although the proposed cage modification is a move in the appropriate direction toward a realistic assessment of the environmental leaching potential of a solid waste, the modification was prematurely proposed. Several commenters declared that the cage modification should not have been proposed before the EPA and ASTM round robin testing has been completed. Other commenters noted that available data are insufficient to determine the impact the proposed change will have on method performance and compliance determinations.

One commenter requested that EPA "hold the proposal in abeyance" for one year for additional testing and evaluation of the proposed change. They suggested that EPA publish a supplemental notice in one year, making additional data available for public review and comment. Along the same lines, another commenter requested that EPA accept additional comments once testing of the cage is completed. The commenter is currently performing tests using the cage; once these are complete they will analyze the resulting data and develop comments and suggestions to be submitted to EPA.

One commenter questioned the validity and reproducibility of the TCLP, as well as specific test requirements such as that for milling or grinding solidified wastes. They claimed that current data are insufficient to evaluate whether the cage tumbling technique would adversely affect the determination of compliance with regulations and treatment standards developed based on TCLP tests using the milling/grinding technique.

The Agency agrees that additional data on performance of the stainless steel cage are needed, and has decided not to go forward with the cage modification at this time.

One commenter, a participant in the joint EPA and ASTM round robin testing of the proposed modification, reported problems with nickel leaching from the stainless steel cage and equipment incompatibility with wastes that evolve gases when in contact with water.

The Agency feels that leaching from the cage may not be a major problem. However, until additional information is available from studies currently under way, the Agency has decided to postpone a final decision on the use of the stainless steel cage.

Several commenters suggested that the new test method may not be truly representative of actual conditions existing in various landfills and waste disposal sites. They were concerned about the unnatural abrasive action of the cage on the wastes. One commenter believed that the tumbling action of the solid sample in the cage could cause particles to be sloughed off by abrasive action and that leaching from these particles would be greater than that of the solid mass of the waste itself. They recommended securing the solid sample within the cage or casting the wastes in the exact shape and size of the cage. Another commenter recommended performing comparative testing for metals on wastes extracted with and without the cage.

The Agency notes that use of the stainless steel cage is not an attempt to replicate any specific landfill conditions. Rather, it is an attempt to give credit to solidification processes and to wastes that are relatively impermeable and structurally stable and are likely to remain so after disposal. The Agency has generally found that wastes that are well solidified maintain their integrity after tumbling in the cage whereas wastes that are poorly solidified break up in the cage. However, the Agency has determined that additional studies on the effect of the stainless steel cage and operational aspects of the procedure are necessary before this proposed modification can be finalized.

Three commenters believed that because the cage's construction provides numerous crevices and a significant amount of surface area for waste residue to collect, an effective cage cleaning method must be developed and specified in the text of the TCLP method.

The Agency agrees that an effective cage cleaning method should be developed. However, since the Agency has decided to postpone a final decision on the cage, a cleaning procedure is moot at this point.

One commenter claimed that the specified cage for the TCLP method is not compatible with most of the commercial rotary agitators currently in service. They stated that it would be "an unnecessary financial hardship to the hundreds of commercial and government labs ... to purchase redesigned agitators to suit the cage modifications as proposed." They also suggested that the free-fall length of the cage be specified as a proportion of sample height, thereby allowing the use of different sized bottles.

The Agency agrees and has changed the free-fall length specification to 9.0 +/- 0.1 inches and the diameter to 3.0 +/- 0.1 inches in order to accommodate a greater variety of bottle sizes. Additional investigations are needed to determine whether this modification will impact the results of the test.

Four commenters were concerned that the shock absorbing springs on the stainless steel cage of the modified TCLP apparatus exert too much force, thereby creating a safety hazard. One commenter recommended that the spring force be reduced by at least 50 percent. On the same note, another commenter reported numerous broken leaching bottles in their studies, which they attribute to the coupling of stresses from the cage springs and the rotary extractor clamp.

The Agency agrees that the force of the springs is excessive and has asked the manufacturers to redesign the cages to exert less force.

Six commenters suggested that freeze/thaw and wet/dry studies do not accurately simulate actual landfill conditions. One commenter contended that wastes may be subjected to colder temperatures in Subtitle C landfills only once when they are first placed in the landfill in freezing temperatures. They also felt wastes will not be subject to 28 wet/dry cycles in a landfill environment. Five other commenters agreed that most hazardous waste is disposed of below the regional and/or average U.S. annual frost penetration; therefore, the test should not be based on freezing and thawing but rather on unconfined compressive strength.

One commenter additionally claimed the waste in the landfill may become wetted with leachate which passes through the disposal unit, but the material will attain an equilibrium level of moisture and will not 'dry out' as assumed in the wet/dry tests.

Another commenter declared that although a monolithic block may form after stabilization, it will eventually disassociate. This commenter stated that "all stabilized wastes will, at one time or another, break-up and disassociate to one degree or another." The waste at this point would come into contact with ground water and a leachate would be formed. Therefore, they believed that Method 1311 should not be changed as proposed.

The Agency responds that it is not known whether or not stabilized wastes will inevitably break up in the landfill environment. Wet/dry and freeze/thaw tests are commonly used to test the long-term integrity of structural materials and it is assumed that the wastes passing these tests will usually remain in a monolithic form under landfill conditions. Additional information on the long term integrity of stabilized wastes is needed.

Five commenters stated that grinding penalizes the solidification industry, and they also believed that monolithic samples that are to be tested for volatile organic constituents (VOCs) should not be milled as part of the procedure. It is generally believed that grinding these wastes results in substantial loss of organic constituents due to volatilization. Thus, accurate analytical determination of these constituents would be impossible. One commenter noted that grinding wastes also penalizes those facilities where wastes are solidified or whose wastes are already in monolithic form.

Two commenters addressed the concern that milling solidified waste does not represent actual conditions that exist in a landfill. These companies urged the Agency to develop testing procedures that would test a waste in the same physical form in which it will be exposed to landfill leaching conditions. The milling procedure would produce material that is no longer representative of the waste that is actually being disposed.

One commenter recommended that alternative designs of the ZHE be accepted, so that the cage approach can be applied to wastes with volatile constituents. They believe that grinding a waste prior to testing in the ZHE will restrict acceptable disposal techniques and penalize confinement techniques of stabilization and encapsulation.

The Agency is not aware of any alternate ZHE design that will accomplish the goals of Method 1311 with minimum volatile loss. If such a design is developed, the Agency will consider it for adoption. Currently, samples to be analyzed for volatile constituents will still have to be ground. When and if the Agency develops or becomes aware of validated procedures for overcoming this problem, changes will be considered for Method 1311.

One commenter recommended that cryogenic crushing of solids be employed. They reported success using a process that coates samples and grinding/milling equipment with liquid nitrogen before processing.

The Agency notes that this is a good technique for particle size reduction and Method 1311 allows its use. However, the high cost involved prevents EPA from requiring its use in all cases.

Another commenter suggested that the Agency use a procedure similar to a method currently being developed by the ASTM D.34 committee for determination of the absorption coefficient of soils. This procedure involves tumbling soils in water containing dissolved volatile organics. The tumbling is performed in bottles with septum caps, and thus no headspace. This allows samples to be withdrawn directly from the bottle using a syringe. An analogous procedure could be used to determine the volatiles leached from solidified wastes. A multi-laboratory validation study has demonstrated that loss due to volatilization is minimized during this procedure.

The Agency considered the use of 40 ml VOA vials with septum caps for the extraction of volatiles from solidified wastes, but decided that it would be very difficult to obtain representative waste samples of the small size that would be required. The Agency also considered the use of the bottle extractor and cage with a septum cap, but determined that it would not be possible to design a bottle that could hold exactly 100 grams of waste and 2 liters of extraction fluid with no headspace for all types of monolithic wastes. Thus, the Agency will continue to require use of the ZHE and grinding of wastes to be analyzed for volatile constituents.

Three commenters indicated that wastes less than 9.5 mm in diameter should not be milled. One commenter urged the Agency to reconsider the requirement to use the cage in the extraction bottles for samples already finely divided (i.e., samples less than 9.5 mm). They noted that for samples already "pulverized" a test of their structural integrity is useless. They also pointed out that this further complicates the procedure and presents an equipment cleaning problem.

Another commenter suggested that a requirement to use the cage with wastes that are already smaller than 9.5 mm is inconsistent with the intent of the cage modification. The cage modification tests the resistance of such wastes to environmental stresses. However, wastes passing through a 9.5 mm sieve without processing are not subjected to such stresses. In addition, it is noted that there is no explanation why wastes undergoing size reduction are extracted without the cage and wastes not requiring size reduction (because they pass a 9.5 mm sieve) should be extracted with the cage in place.

The Agency generally agrees and will address these comments when and if it proceeds with the cage modification.

Seven commenters declared that the requirement that encapsulated wastes be ground or milled is inappropriate. Specifically, one commenter recommended that encapsulated wastes be pre-formed into cylinders or blocks to fit the extraction cage as is proposed for fixed or stabilized wastes. Other commenters indicated that EPA has not adequately evaluated all encapsulation techniques prior to the proposed modification.

The Agency feels that until sample integrity and leaching prediction improve, modifications to Method 1311 must be conservative. The Agency welcomes additional information regarding encapsulation techniques. Modifications will be made to the procedure as additional data become available and justify the change.

One commenter suggested that encapsulation techniques vary greatly in the degree of confinement. Radioactive waste, for example, is encased in a binder such as cement as opposed to a thin protective coating. They noted that, in instances such as this, the waste is similar to a stabilized monolith and should be treated as such. Another commenter suggested that the Agency should allow materials that it considers "stable, non-corrodible, encapsulated wastes" to be extracted without size reduction. They indicate that these wastes could be visually distinguished from those in corrodible shells. A third commenter declared that the requirement to reduce the size of encapsulated waste negates the purpose of encapsulation (i.e., to prevent the waste from coming into contact with landfill leachate). They agreed that milling products having the potential for internal corrosion (e.g., batteries) is reasonable, but recommended that vitrified or polymer encapsulated wastes be exempted from the size reduction protocol.

The Agency agrees that encapsulation techniques vary greatly in their degree of confinement. EPA is currently working with the Department of Energy (DOE) in developing a definition for encapsulation that will distinguish between those wastes that are non-corrodible and stable from those that are corrodible.

In response to EPA's invitation for comment on how to define "stable, non-corrodible, and encapsulated" wastes, one commenter suggested that the Agency follow the precedent that has been established by the Nuclear Regulatory Commission (NRC). This commenter indicated that existing NRC criteria for radioactive wastes appear to be applicable for other wastes that will be land disposed. They noted that EPA's use of current definitions would eliminate the need for duplicate efforts and improve regulatory consistency among Federal agencies.

The Agency agrees with this commenter that defining "stable" in the manner found in 10 CFR Part 61 (Stable: Maintaining gross physical properties and identity) may be a reasonable approach, however it lacks any time component. The remaining two definitions, "non-corrodible" and "encapsulated," were not found to be referenced in 10 CFR Part 61. The following are some preliminary draft concept definitions under consideration.

> Non-corrodible: Able to survive in the environment over an extended period of time without chemical change in exposed surfaces.

> Encapsulation: Application of a surface coating through coating/sealing, or placement in specially designed/sealed containers, which provides a surface barrier that prevents contact between the waste and leaching fluids that may be present in the environment and that exhibits long-term stability.

One commenter indicated there is a discrepancy in distinguishing between stabilized monoliths and various types of encapsulated wastes. They noted that High Integrity Containers, designed to survive in the environment for a minimum of 300 years, provide containment similar in concept to the liners required for hazardous waste disposal facilities. Encapsulation techniques vary greatly in the degree of confinement provided and, therefore, should be evaluated on an individual basis and differently from stabilized wastes.

The Agency agrees that encapsulated and stabilized wastes are very different and EPA is investigating different approaches to regulating these types of wastes. However, sufficient information on the behavior of these materials in landfills is not yet available.

One commenter claimed that the term "equivalent material," with regard to the construction material for the cage, should be better defined. They believed that criteria used in judging equipment equivalence (e.g., structural strength, chemical inertness) should be specified. This would allow the users of the test to rationally judge the acceptability of cage designs other than those constructed of 100 percent stainless steel.

The Agency feels the term "equivalent material" is satisfactory and needs no further explanation. Equivalency is further defined in the Agency's hazardous waste methods Equivalency Guidance Manual. ("Test Method Equivalency Petitions: A Guidance Manual"; EPA/530-SW-87-008; OSWER Policy Directive Number 9433.00-2, February 1987.)

One commenter suggested that EPA should not modify the TCLP as proposed by eliminating the requirement for particle size reduction. The commenter declared that the proposed modification to the TCLP would increase the threat of pollution from landfill leachate and would encourage accelerated landfill utilization and expansion with no environmental gain.

The Agency responds that there is not enough information to determine the extent to which the proposed modification would effect waste characterization results. For example, work reported at the 1988 Symposium on Waste Testing and Quality Assurance (Prange and Garvey, 1988) shows that larger particles may actually leach more readily than smaller particles. Experiments with two specific cement-solidified wastes demonstrated that arsenic and chromium were leached more effectively from large rather than small waste particles by the TCLP extraction. As previously stated, the Agency has decided not to go forward with the proposed cage modification at this time due to insufficient evaluation and the potential problems experienced with leaching of nickel and chromium, cleaning the cage, and some other operational difficulties.

One commenter referred to comments submitted on the previous proposals regarding the TCLP, and repeated their opposition to the proposed rulemaking and the "TCLP method per se."

The Agency addressed comments of this nature in other sections of this background document.

One commenter suggested changes in two sections of the flowchart addressing particle size reduction.

The Agency disagrees with the suggested changes because they deviate from standard flowchart format found in the guidance manual "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, and provide no additional technical guidance.

One commenter agreed that EPA is correct in its conclusion that the cage modification would not have an economic impact resulting in annual expenses exceeding $100 million.

## VII. Analytical Constraints

The regulatory levels for the compounds proposed for inclusion in the expanded Toxicity Characteristic span about 5 orders of magnitude (i.e., from the low parts per billion to 100 parts per million). This is not so much a function of the individual dilution/attenuation factors but is due instead to the great range in toxicity levels of the individual toxicants. Since many of the toxicity levels for the carcinogens (and some of the non-carcinogens) are very low, the calculated regulatory threshold will also be very low (depending on the magnitude of the dilution/attenuation factor). The proposed regulatory thresholds were below the practical quantitation limits (PQL) which can be measured with current methodology for seven compounds. Due to changes in the chronic toxicity reference levels and the dilution/attenuation factors, the final regulatory levels are below the PQL's for three compounds (2,4-dinitrotoluene, hexachlorobenzene, pyridine).

The Agency received eighty-two comments which address the topic of analytical constraints on the use of the TCLP in the Toxicity Characteristic. These comments came from consultants, research laboratories, petroleum/chemical companies, trade associations, state and federal agencies, municipal sewer authorities, waste management companies, electric utilities, and other industries. The comments were grouped into three areas: use of quantitation limits as the regulatory level for some compounds; analytical methods in general; and SW-846 Method 8270.

### A. Use of Quantitation Limits

EPA proposed to deal with calculated regulatory thresholds which fall below the analytical detection limit by establishing technology-based regulatory levels. The lowest contaminant concentration that can be reliably measured within specified limits of precision and accuracy during routine laboratory operating conditions is the quantitation limit. The quantitation limit represents the lowest level of measurement which can be achieved by good laboratories within specified limits during routine laboratory operating conditions. The quantitation limit is determined through inter-laboratory studies (i.e. performance evaluation studies).

If data are not available from inter-laboratory studies, quantitation limits are based upon the method detection limits and an estimate of a higher level which would represent a practical and routinely attainable level with relatively high certainty that the reported value is reliable. EPA proposed using a value of five times the analytical detection limit as the quantitation limit and proposed setting the regulatory level at the quantitation limit for those compounds for which the calculated health-based threshold falls below the quantitation limit.

Some comments supported EPA's proposal. Two stated that, when detection limits are a problem, the samples could be concentrated to as low as 100 microliters from the EPA-specified 1-mL samples (to increase sensitivity by a factor of 10). Gas chromatography program rates can also be changed, in some cases, to optimize the sensitivity for a particular compound.

The Agency responds that, when semi-volatile compounds are the analytes of interest, concentration of the sample to a 100 microliter volume may result in the loss of significant amounts of the analytes. GC program rates are optimized to produce certain results, and changes to the program may involve a trade-off in that sensitivity to other analytes may be compromised. The reproducibility of the analysis may also be affected. Therefore, the Agency does not recommend changing the GC program rates.

Several comments gave qualified support for the use of quantitation limits defined as five times the detection limit for the compounds with health-based thresholds below this level. One asserted that the detection limits listed in the proposed rule apply to water samples only and may not be applicable to more complex matrices. Other comments also expressed concern about whether or not these detection limits apply to waste samples encountered in the RCRA program. One stated that the use of five times the detection limit observed in water is not appropriate for samples containing filterable oil, since the liquid oil phase must be analyzed for the contaminants. Analyzing non-aqueous phases usually results in increased detection limits relative to water because of the severe dilutions that are associated with the digestion procedures used on oil samples.

All of these comments urged EPA to determine actual quantitation limits of the regulated compounds in real wastes and establish a procedure to handle situations where quantitation at the regulatory level is not possible. One comment stated that, if an analytical level of five times the detection limit is not achievable in real waste extracts, the analytical level should be determined by extracting the waste after the waste has been spiked with substances of interest. The analytical level could then be established on a case by case basis. The method of standard addition could also be used in such cases. Also, one comment recommended that EPA should state the calculated health-based threshold levels for compounds with higher detection or quantitation levels. This would eliminate the need for amending the regulatory level for a compound if the analytical methodology improves. Another comment stated that compound-specific evaluation is necessary to insure that regulatory levels are not unreasonably high for extremely toxic constituents.

Many comments criticize the proposed quantitation limits. Several comments indicated that the proposed limits may not be technically and/or economically achievable for specific wastes. One comment stated that it is inappropriate to use the quantitation limit observed in water for waste extracts and filtrates, and noted that experience with actual waste leachates has shown that these are complex matrices in comparison with water. This comment recommended that quantitation limits be determined on a case-by-case basis to account for the varied matrices, and that EPA specify definitions and calculation methods for method detection limits and quantitation limits.

Other comments also stated that it is inappropriate to equate the quantitation limits observed in water with that of the TCLP extract. The TCLP extract will contain many contaminants derived from real wastes that cause analytical interferences and the TCLP leaching fluid itself contains a significant concentration of acetic acid. This comment recommends utilizing the existing contract required detection limits (CRDLs) for medium soil/sediment available from the EPA Contract Laboratory Program (CLP) for estimating the quantitation limits for real wastes.

Two comments stated that EPA's selection of regulatory limits is premature and arbitrary. These commenters stated that the MDL is only an estimate and that the values listed in the Federal Register may be found to be inappropriate when the actual waste extracts are analyzed. One suggested that the table showing detection and quantitation limits should be altered to show either the detection and quantitation limit for each mode of detection or for the least sensitive form of measurement. This comment stated that whatever modification is made, the detection and quantitation limits should be consistent and scientifically correct.

Twenty-four comments supported alternatives to the proposed quantitation levels. Some supported higher quantitation limits, while others supported the use of detection limits or other alternatives.

Nineteen comments supported higher quantitation limits than those proposed by EPA. Some of these suggested a factor of 10 times the analytical detection limit as the defined quantitation limit, while a factor of 25 was recommended by others. Still other comments suggested that a factor of 10 be used for relatively clean sample matrices, but for more complex matrices (especially nonhomogenous, viscous, oily, or otherwise more analytically intransigent solids) a factor of 25 times the detection limit should be used. One suggests adopting the approach described by the American Chemical Society's "Principles of Environmental Analyses" Anal. Chem. 1983, pp. 2210-2218. All of these comments indicated that such changes are necessary because of problems inherent in the analyses and because of the variability of accuracy and precision even under ideal laboratory conditions found in inter-laboratory studies.

The Agency agrees that the ability to achieve the quantitation levels listed in the proposed rule depends strongly on the type of waste which is being analyzed. It is recognized, however, that determination of a matrix dependent quantitation limit would require analysis of a variety of wastes. EPA feels that it is impractical to perform waste-specific analyses to investigate the effect on quantitation limits at this time. Therefore, EPA has chosen to use five times the method detection limit as the quantitation limit.

Another comment stated that setting the quantitation limit at 5 times the detection limit is unreasonable for detection limits of less than 10 ppb, since analytical variability in this concentration range is greater than at higher levels. This comment suggested that a sliding scale tailored to the detection limit would be more appropriate. Such a scale should use a factor of 10 for materials with a detection limit of 1 ppb or less and a variable factor starting at 10 and ranging down to a factor of 5 at 10 ppb or above. This would allow for the greater variability inherent in measurements of very low concentrations.

The Agency notes that the compounds for which the health-based threshold is less than five times the detection limit have detection limits of 10 ppb or greater.

Three comments supported the use of the analytical detection limits as the regulatory threshold. These comments stated that, for carcinogens, the use of a quantitation limit is inappropriate. The goal is to achieve the safest environmental conditions, and the detection limit represents the lowest achievable measurement (even if not consistently achieved). Therefore, using the analytical detection limit would be more protective of human health and the environment, and will have the dual effect of somewhat lowering the cancer risk posed by these carcinogens and encouraging laboratories to develop better analytical capabilities. One comment recommended that EPA pursue the development of more sensitive analytical methods that would allow the quantitation of these carcinogens at the $10^{-6}$ risk level.

The Agency responds that it is constantly striving to improve the sensitivity of analytical methods in order to provide increased protection for human health and the environment. Analytical detection limits are, by definition, not routinely achievable under average laboratory conditions. Thus, a regulatory level set at the detection limit would be difficult to enforce and difficult for the regulated community to demonstrate compliance. In order to provide a consistently enforceable regulatory limit, the Agency has decided to define the quantitation limit as five times the detection limit and to set the regulatory level for the five compounds at the quantitation limit.

Another comment favored establishing regulatory levels which are based on treatment technology rather than measurement technology. This comment also noted that any change in the regulatory level established in the regulation must be subjected to the rule-making process and not automatically based on improved detection limits or treatment technology.

The Agency responds that the Toxicity Characteristic is a health-based regulation, not a technology-based regulation. The Agency agrees that changes in levels are subject to the federal rule-making process.

One commenter expressed concern that the action levels proposed by EPA are below the ability of existing analytical procedures to detect, and therefore the commenter would be unable to demonstrate that its waste (specifically, landfill gas condensate) is non-hazardous.

The Agency points out that this comment demonstrates a misunderstanding of the proposed rule. In both the proposed and final versions of the Toxicity Characteristic, when the calculated regulatory threshold for a pollutant close to the analytical detection limit, the threshold is set at 5 times the detection limit, defined as the quantitation limit. Therefore, if a generator can demonstrate that the concentration of regulated substances in the TCLP extract is below the quantitation limit, the waste is non-hazardous according to the Toxicity Characteristic.

## B. Analytical Methods - General

The test methods proposed for analysis of TCLP extracts for compliance with the Toxicity Characteristic were listed in the Federal Register (Friday, June 13, 1986, pp. 21672-21673, Table C-2). These methods are contained in EPA publication SW-846, "Test Methods for Evaluating Solid Wastes".

One comment supported EPA's proposed analytical methods, stating specifically that GC/MS should be used for identification and quantitation of organic compounds since GC/MS is a definitive method which will facilitate the enforcement of the proposed rule.

Nineteen comments gave qualified support for the proposed analytical methods. Many expressed concern over the relatively small number of laboratories which can perform the TCLP and associated analyses. The consensus was that the high cost and specialized nature of these tests would force most customers to use commercial laboratories that are participating in EPA's contract laboratory program under Superfund. These laboratories already have high sample loads, and additional work will result in substantial delays, high prices, or both.

The Agency replies that independent laboratories and the regulated community have had sufficient time to prepare to perform the TCLP since its proposal on June 13, 1986. The method is already required for demonstrating compliance with the Land Disposal Restrictions Rule promulgated on November 7, 1986 (51 FR 40643-40652). In addition, the modifications to the Toxicity Characteristic will not take effect until six months after publication of the final rule in the Federal Register.

One comment stated no specific objections to the analytical methodology in the proposed rule. However, the revised SW-846 method manual should be made available before the rule is finalized.

The Agency notes that the Third Edition of SW-846, "Test Methods for Evaluating Solid Waste-Physical/Chemical Methods", was announced in the Federal Register on March 16, 1987 (52 FR 8072). It is now available through the U.S. Government Printing Office, Washington DC, 20402, Publication Number 955-001-00000-1, at a cost of $110, which includes future updates.

Three comments stated that equivalent EPA approved methods (which may be more cost effective and provide the same accuracy) should be allowed. For example, an alternative to GC/MS for volatiles is purge and trap gas chromatography with tandem detectors (such as the Hall detector for chlorinated volatiles and the photoionization detector for volatile aromatics). This essentially combines methods 8010 and 8020. A separate portion of the extract could be purged and trapped on a gas chromatograph using a flame ionization detector for those compounds for which neither the halide specific detector nor the photoionization detector is applicable. Similarly, semi-volatile chlorinates can be analyzed with Method 8120.

The Agency agrees that several methods are available to conduct the required analyses. Methods approved for demonstrating compliance with the Toxicity Characteristic appear in SW-846, and the analyst may choose from among these methods when performing the analysis. Additional methods may be submitted to the Agency for review and evaluation, and may be included in future editions of SW-846.

Two comments made suggestions for dealing with potential analytical problems. Since interferences with halogenated compounds or background problems are a problem on an ECD, capillary columns, rather than packed columns (Method 8080), should be used for the pesticides. Also, identification should be used for the pesticides. The identification should be verified by GC/MS where the concentration permits.

The Agency notes that, in the revised SW-846 manual, alternative gas chromatography columns are allowed if appropriate quality control procedures are followed. Method 8000 (Gas Chromatography) discusses equipment, procedures, and quality control for all GC analyses.

Six comments criticized the proposed analytical methods. Some concern was expressed over the high variability of the SW-846 methods. One comment stated that either the analytical methods are being pushed to their limits of being able to accurately quantitate the components in the extract or there is a persistent problem with matrix interference. Two comments supported and encouraged EPA to revise the analytical methods based on rigorous interlaboratory testing and validation.

The Agency states that the Third Edition of SW-846 contains additional method performance data. SW-846 methods are comparable in performance with analytical methods used in the water and wastewater program and the Superfund program.

The fact that atomic absorption methods are unable to distinguish among the various forms of chromium and therefore only measure total chromium was a concern of one comment.

The Agency is aware of the analytical problems associated with chromium. For this reason it regulates total chromium, and is continuing to investigate alternative analytical procedures to distinguish between the various forms of the element.

Finally, one comment stated that, since it seems apparent that the Toxicity Characteristic list will be expanded to include a broadened spectrum of organic compounds, EPA should actively seek to establish reliable and cost effective indicator analyses that can be used to screen for classes of compounds that are of concern. GC/MS analyses may not be appropriate in all cases when the true concern should be a less rigorous and more cost effective measure of the degree of hazard posed by a waste.

The Agency agrees that reliable and cost effective indicator analyses are desirable. Research is currently being conducted in this area. New methods will be announced for public comment when available, and included in SW-846 when validated.

### C. SW-846 Method 8270

As discussed in the preamble to the proposed rule, analyzing the TCLP extract for phenolic compounds and phenoxy acid herbicides poses a potential analytical problem. The leaching fluid used in the new leaching procedure is 0.1 M with respect to acetate. Because of interference from the acetate ion, the analytical method most frequently used for these compounds (i.e., GC/MS SW-846 Method 8270) may not suffice. EPA is currently investigating these methods to ascertain whether they are sufficient or whether it may be necessary to modify these methods. One modification being investigated is whether it may be possible to remove the acetate ion from the extract before determination of the phenolics and herbicides.

EPA is also investigating the use of high pressure liquid chromatography (HPLC) using electrochemical and fluorescence detection. HPLC with fluorescence detection was used in developing the improved leaching procedure and has been shown to produce acceptable results. A GC/MS method would be preferable since using the HPLC method could add significantly to analytical costs. Should the presence of the acetate ion present substantial problems to GC/MS, HPLC may be specified.

Five comments presented evidence supporting removal of the acetate ion from the extract before determination of the phenolics and herbicides using GC/MS. Two comments address the complications caused by the use of acetic acid in the leaching procedure. Whereas the problem is greatest with the phenols and cresols, another step is needed even for the nonionizable species to eliminate the acetate from the methylene chloride extract. Analysis by EPA Method 8270 may lead to the destruction of the GC column. Without the ability to analyze the acid fraction, a number of environmentally significant (and water soluble) compound classes (i.e., phenols) will be overlooked. Development of a method such as reverse phase open column cleanup was recommended in one comment as a means for dealing with the acetate ion. Another comment asserted that the analysis of the extract for acid compounds by GC methods will be impossible because of the acetic acid matrix interference.

The Agency agrees that analysis for acidic compounds by GC methods may be difficult. The use of a bonded-phase capillary column can reduce the interference from acetate. Methods for acetate removal are still under investigation. The Agency welcomes alternative suggestions, especially when accompanied by supporting data.

Ten comments addressed alternatives to GC/MS. Three of these believe that HPLC should not be recommended because of its higher cost and lack of availability. Three others indicated that GC/MS was a better method than HPLC. One stated that there is no description of how the HPLC method would be implemented. At the very least, a lengthy verification process would be required. Even if a good HPLC cleanup is approved, use of the method is bound to increase the analytical costs and slow down the analytical throughput.

The Agency agrees that the specificity of GC/MS analysis is more advantageous than the HPLC, in despite of the associated difficulties. HPLC methods for phenols are not included in the Third Edition of SW-846 due to the lack of validation data.

Four other comments stated that such a major rule should not be proposed while EPA is uncertain about how the analysis should be performed. It was recommended that EPA investigate the need to monitor phenols and cresols and determine if such monitoring is premature, considering the associated analytical problems. One commenter opposes establishing a regulatory level for cresols until the analytical problems are resolved.

The Agency replies that it is possible to analyze for total cresols, while it is not possible to separate the "m" and "p" isomers. For this reason, the regulatory level has been established for total cresols ("o", "m", and "p" isomers).

One final comment stated that, due to the limited ability of GC/MS to handle polar compounds, the Agency is considering using a less specific nondefinitive method (i.e. HPLC with either electrochemical or fluorescence detection). This mode of detection is not specific enough for analysis in enforcement situations. A better approach would be thermospray/HPLC/MS (TSP-LC/MS) for the following reasons:

- Provides definitive and specific results for polar compounds;
- Consistent with previous Agency choice of definitive analytical methods;
- No significant added expense for implementing the HPLC;
- Eliminates costly false positive values;
- TSP-LC/MS are commercially available and can be easily retrofitted to most instruments; and
- Electrochemical and fluorescence detectors have little specificity and are subject to interferences from other chemical species.

The Agency notes that thermospray is very expensive and that such a method has not yet been standardized. The EPA welcomes the submission of performance data for alternative methods.

## VIII. Changes to the TCLP

### A. EPA Changes Made Prior to Review of Public Comments

EPA has identified a number of technical and procedural changes to the TCLP based on its own further evaluation of the method. Specific changes are noted below. Section numbers refer to the proposed method (51 FR 21685-21691). Section numbers in the final version of the method appear in parentheses.

2.3 This step is changed to say "If compatible (i.e., multiple phases will not form on combination)..." The change reflects the deletion of the formation of a precipitate as condition of incompatibility.

4.2.1 Add: For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

4.2.1 Add: The ZHE should be checked after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

4.2.2 Add: It is recommended that borosilicate glass bottles be used over other types of glass, especially when inorganics are of concern. Plastic bottles may be used only if inorganics are to be investigated.

4.6 Add instructions on choosing a collection device to be used with the ZHE as follows:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of non-aqueous liquid (i.e., <1% of total waste), the TEDLAR bag should be used to collect and combine the initial liquid and solid extract. The syringe is not recommended in these cases.

4.6.2 If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

7.0 - 7.5 (New Section on Preliminary TCLP Evaluations) This Section is for determining both percent solids and the extraction fluid (to be used in the procedure where volatiles are not involved). This determination is performed on a separate sample. Once this determination has been performed, another sample taken earlier is used for the next procedure. The procedure which is used at this point depends on whether or not the sample contains volatile contaminants.

7.0 (8.0) Replace 75g sample requirement with "Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by the performance of a single TCLP extraction will not be sufficient to perform all of the analyses to be conducted, it is recommended that more than one extraction be performed and that the extracts from each extraction be combined and then aliquoted for analysis.

7.6 (8.6) Add: Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

8.0 (9.0) Add: Any manipulation of the waste, the extract, or the initial liquid phase should be done while cold (i.e., 4 degrees C), to minimize loss of volatiles.

8.12 (9.13) Change the last sentence to read: All extract shall be filtered and collected if the TEDLAR bag is used, if the extract is multi-phasic, or if the waste contained an initial liquid phase (see Steps 4.6 and 9.1).

(10.3) Addition of the matrix spike should occur once the TCLP extract has been generated. (i.e., should not occur prior to the performance of the leaching step).

9.5 (10.6) Add: Samples must undergo TCLP extraction within the following time period after sample receipt: Volatiles, 14 days; semi-volatiles, 40 days; Mercury, 28 days; and other metals, 180 days. Extraction of the solid portion of the waste should be initiated as soon as possible following initial solid/liquid separation.

## B. Changes Made to TCLP as a Result of Public Comments on the June 13, 1986, Proposal

EPA has made additional technical and procedural changes to the TCLP as a direct result of the public comments. These changes are listed below.

2.2 The first sentence of this step is changed to: "For wastes comprised of solids or for wastes containing significant amounts of solid material, the particle-size of the waste is reduced (if necessary). The liquid phase, if any, is separated from the solid phase and stored for later analysis."

4.2.1 Add: This device is for use only when the waste is being tested for the mobility of volatile constituents (see Table 1).

4.3 Add: It is recommended that all filtrations be performed in a hood.

4.3.2 Add to the description of the filter holder: Vacuum filtration is only recommended for wastes with low solids content (<10%) and for highly granular (liquid containing) wastes. All other types of wastes should be filtered using positive pressure filtration.

4.3.3 Add: Devices made of high-density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used only when evaluating the mobility of metals.

5.1 Changed to Reagent Water, which is then defined.

5.5 Glacial acetic acid (HOAc) ACS Reagent grade

5.6.2 Change NOTE to: It is suggested that these extraction fluids be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately.

6.2 New Section: At least two separate representative samples of a waste should be collected. If volatile organics are of concern, a third sample should be collected. The first sample is used in several preliminary TCLP evaluations (e.g. to determine the percent solids of the waste; to determine if the waste contains insignificant solids (i.e. the waste is its own extract after filtration); to determine if the solid portion of the waste requires particle size reduction; and to determine which of the two extraction fluids are to be used for the non-volatile TCLP extraction of the waste).

6.4 (6.5) Change "care must be taken to ensure these (i.e., volatiles) are not lost" to "care should be taken to minimize the loss of volatiles."

7.0 - 7.17 Becomes 8.0 - 8.15: Procedure when volatiles are not involved

7.7 (7.1.7) Add: NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in step 7.1.5 to determine the weight of the waste sample that will be filtered.

7.10 (7.2.2) Add: Until two successive weighings yield the same value within + or - 1%.

7.10.2 (7.2.2) Add NOTE: It is recommended that the drying oven be vented to a hood or appropriate device.

7.13 (8.9) Change to: Quantitatively transfer the solid material into the extraction vessel, including the filter used to separate the initial liquid from the solid phase.

7.13 (8.11) Change the length of time of the extraction from 18 hours to 18 + or - 2 hours. Indicate that the temperature "22 + or - 3 degrees C" is air temperature.

7.14 (8.12) Add: For the final filtration of the TCLP extract, the glass fiber filter may be changed if necessary to facilitate filtration.

7.16 (8.14) Change to: The extract should be immediately aliquoted for analysis and properly preserved. (Metals aliquots must be acidified with nitric acid to pH <or=2; all other aliquots must be stored under refrigeration (4 degrees C) until analyzed).

8.0 - 8.15 Procedure when volatiles are involved becomes 9.0 - 9.16.

8.0 (9.0) Add: The ZHE is charged with sample only once and the device is not opened until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Add: The ZHE device is to be used to obtain TCLP extracts for volatile analysis only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes (e.g. metals, pesticides, etc.).

Add: Definition of solids as "that fraction of a sample from which no liquid may be forced out by an applied pressure of 50 psi".

8.2 (9.2) Add: Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample.

8.5 (9.6) Add: The means used to effect particle size reduction must not generate heat in and of itself.

8.14 (9.15) Change - "determine the volume of the individual phases (to 0.1 ml)" to "determine the volume of the individual phases (to + or - 0.5%)."

9.0 - 9.5 Moves to Section 10.0 - 10.6.

9.2 (10.2) Add specification that extraction fluid #1 is to be used for the method blank.

9.4 (10.5) Change "for each waste type" to "for each analyte".

TCLP Flow Chart - Add arrowheads.

C. Changes Proposed in May 24, 1988, FRN

Section numbers refer to the TCLP (Method 1311) as finalized for use in the Land Disposal Restrictions program on November 7, 1986, 51 FR 40643-40652.

Figure 1 - Flow chart has been revised

Table 2 - Environmental Machine and Design, Inc., and Millipore Corp. are added as suppliers of rotary agitations apparatuses.

Table 3 - Lars Lande Mfg. is added as a supplier of Zero Headspace Extrator Vessels.

Table 5 - Millipore and Nucleopore are added as suppliers of suitable filter material.

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